

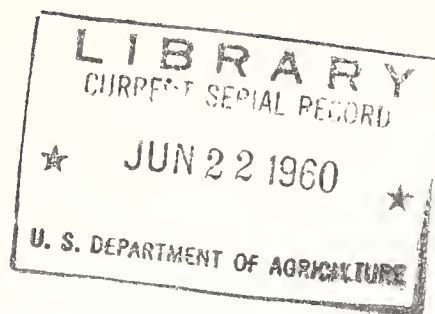
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# AN INVESTIGATION OF INORGANIC SALTS AND OXIDES AS FINISHES FOR COTTON

Agricultural Research Service  
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## *Abstract*

A search was made of the literature pertaining to the treatment of cotton with inorganic compounds to improve certain functional properties. From approximately 700 references, which purportedly related to this subject, 310 were selected as having some pertinency to the problem. These are cited in the text around certain specific topics and are arranged in alphabetical order in the Bibliography.

A systematic screening of oxides and salts of metallic elements as finishes which might improve certain of the functional properties of cotton was made. Compounds were selected from various groups of the Periodic Table of the Elements. Seventy-four compounds, selected on the basis of their apparent insolubility, were applied to cotton, some of them in more than one concentration. A battery of tests was employed to evaluate the treatments. The treated fabrics were tested for: Stability to leaching and laundering; effect on strength; changes in resistance to mildew and rot (soil burial), to outdoor weathering, to actinic degradation, to heat, and to burning; changes in thermal stability; and changes in water repellency. Several compounds which had been studied by previous investigators were included for comparative purposes.

From the several compounds studied upon which previous findings had not been reported, the following nine were selected for further consideration: Aluminum hydroxide, beryllium oxide, cobaltous hydroxide, cobaltous metaborate, lead hydroxide, manganous oxide, manganous phosphate, manganous silicate, and nickelous phosphate. These offered promise of improving certain end use characteristics of cotton fabrics.

Outstanding among the nine compounds were cobaltous metaborate and cobaltous hydroxide. Cobaltous metaborate treated cotton was good in mildew and rot resistance, in outdoor weathering performance, in stability with respect to actinic exposure, and in resistance to atmospheric contaminants, and was good to excellent in water repellency. The cobaltous hydroxide treated cotton was found to be good to moderate in resistance to mildew and rot and very good in outdoor weathering. It also imparted good resistance to actinic degradation and to atmospheric contaminants.

# PART I—SURVEY OF THE LITERATURE

by

**Georgia Ellen Wright and Ramon M. Esteve, Jr.**

## INTRODUCTION

## LITERATURE FINDINGS CONCERNING SPECIFIC PROPERTIES IMPARTED TO COTTON

Mildew and Rot Resistance

Weathering

Flame Resistance

Water Repellency

Miscellaneous Properties

## LITERATURE FINDINGS CONCERNING CERTAIN COMPOUNDS APPLIED TO COTTON



# INTRODUCTION

It is recognized that a considerable number of inorganic substances can influence the important textile properties of cotton fabrics. Many such treatments may have been overlooked because no systematic study of inorganic compounds has been undertaken for this purpose. The plan of this therefore, was to conduct a literature survey to ascertain the information available; to select a group of inorganic substances from various sections of the Periodic Table—some of which were and some of which were not mentioned in the literature—to determine whether or not their position could indicate their usefulness in treating cotton for desirable properties; and to evaluate, generally in a preliminary way, the most promising compounds uncovered by the survey.

Approximately 700 literature references reporting on the use of inorganic compounds in the treatment of cotton were reviewed. From these references, 310 were found to discuss (a) the formation of insoluble deposits inside the cotton fiber to enhance such properties as water repellency, weathering stability, resistance to actinic degradation, mildew and rot resistance, and fire resistance; (b) the application of insoluble organo-metallic chelates to improve or provide rot or mildew resistance; (c) the formation of insoluble metallic soaps to produce water repellent and mildew resistant finishes; (d) the application of pigments and resins to produce increased stability by means of reduced accessibility; (e) products forming compounds or chelate formations with cotton to improve mildew and flame resistance; and (f) the application of insoluble organo-metallic compounds or polymers to enhance mildew resistance, water repellency, and flame resistance. These references were particularly pertinent to the problem; they are, therefore,

appended to this report.

Most of the data on inorganic compounds indicated that they were applied as an insoluble deposit inside and on the surface of the fiber. In some instances they were applied in pigment form by use of a resin binder. In a very few cases compound or chelate formation with the cellulose itself was reported. An example of a reported linkage with cellulose is that formed with titanium tetrachloride, and evidence supporting this belief is shown. Other than having flame resistance, cellulose thus treated is found to display mildew resistance (107, 226, 231).<sup>1</sup> Since titanium dioxide is not toxic (252), the mildew resistance can be explained by proposing a reaction with the cellulose, with a protection given in the same manner as that involved in phosphorylated and acetylated cellulose (101). It should be noted that when bonds of modified cellulose are subjected to hydrolysis an insoluble system results which has a greater stability than a homogeneous solution.

The formation of chelates with cellulose has been reported. Cellulose which has been treated with a calcium salt displays a greater thermal stability than untreated cellulose (144). The chelate in this case was reported as being formed by the oxidation of the 6-hydroxy groups.

A series of chemically modified cottons form salts with ions (125), e.g., phosphorylated, amino ethylated, sulfoethylated, and carboxymethylated fabrics. For example, copper salts of carboxymethyl cellulose have been used to impart rot resistance (255). The durability of these finishes, however, would be limited since all of these compounds are subject to ion-exchange.

## LITERATURE FINDINGS CONCERNING SPECIFIC PROPERTIES IMPARTED TO COTTON MILDEW AND ROT RESISTANCE

Biological degradation of cellulose is due to the growth and reproduction of micro-organisms capable of using the fiber as food. Some fungi and bacteria belong to the group which

attack cotton. These organisms secrete enzymes which merely digest the cuticle and permit them to penetrate the secondary cellulose wall. That cellulose is converted to a linear

<sup>1</sup> Numbers in parentheses refer to Bibliography at end of this report.



polysaccharide, with another enzyme hydrolyzing the chain to glucose, has been proposed as a theory to account for the great decrease in breaking strength of the degraded cotton fabrics when there is only a small decrease in the molecular weight (260). Another explanation is that only minute areas are broken down, resulting in only a small change in the degree of polymerization (58).

The subject of mildew resistance has been reviewed by various investigators (28, 51, 57, 113, 115, 177, 260).

Mildew resistance is reported as improved by the following general types of compounds:

- a) Antiseptic compounds that destroy micro-organisms.
- b) Compounds capable of inactivating enzymes. Many references pertaining to this type of treatment of cotton exist in the literature (28, 51, 58, 113, 115, 117, 177). It should be noted, however, that inorganic compounds are not included.
- c) Treatments which modify the cellulose chemically. Micro-organisms are no longer capable of using the altered fiber as food.

The rot and mildew resistance of cotton treated with certain titanium compounds gives evidence of the formation of titanium-oxygen bonds in cotton, as previously noted. A number of organic modifications of cotton impart mildew resistance, for example, acetylation (101), phosphorylation (78, 114, 138), carboxymethylation, and cyanoethylation. Tetrakis (hydroxymethyl) phosphonium chloride also belongs to this group (227, 228, 229).

- d) Compounds that coat the cotton fiber. The coating of fibers has been proposed as another mechanism for decreasing accessibility of cotton to micro-organisms. White and Siu (299) proposed this theory for the observed resistance displayed by urea-formaldehyde treated cotton (51). On the other hand, Dean (54) described cotton treated with other polymers which displayed little resistance to weathering. Cameron and Morton point to crosslinking and reduced swelling as the factors responsible for

the observed resistance to mildew (40). According to Siu (260) the amorphous parts are more accessible to attack. The deposition of insoluble deposits inside the fibers also may be a factor.

## WEATHERING

The weathering of cotton fabrics involves the degradation of cellulose by chemical and biological processes (54, 55, 259). The chemical reactions include oxidative and hydrolytic degradation. The causative agents involved in these reactions include the oxygen in the atmosphere, moisture, light, heat, and gaseous contaminants (consisting of oxides of nitrogen and sulfur, hydrogen sulfide, and a variety of other compounds).

Another important factor in the weathering of cotton fabrics is the autoxidation of cellulose. In the absence of light and heat, the reaction is negligible; but the presence of these factors greatly increases the rate of degradation. This degradation results in decreased viscosity and an increase in the copper number and methylene blue absorption of cotton. A mechanism typical of the oxidation of organic compounds is given below for cellulose.

### Initiation and Autocatalysis

1.  $R_{\text{cell}}H + O_2 \longrightarrow R_{\text{cell}}OOH$
2.  $R_{\text{cell}}OOH \longrightarrow R_{\text{cell}}O + OH$
3.  $R_{\text{cell}}O + R_{\text{cell}}H \longrightarrow R_{\text{cell}} + R_{\text{cell}}OH$

### Chain Reaction

4.  $R_{\text{cell}} + O_2 \longrightarrow R_{\text{cell}}OO$
5.  $R_{\text{cell}}OO + R_{\text{cell}}H \longrightarrow R_{\text{cell}}OOH + R_{\text{cell}}$

### Termination

6. The reaction of two radicals.

Moisture increases the rate of reaction by accelerating the consumption of oxygen (69, 289), as well as autocatalysis, Steps 1 and 2.

The action of heat below pyrolytic temperatures increases the rate of oxidation (48, 274, 289), by accelerating Steps 1 and 2. In this connection, heat aging was investigated by Conrad (48, 274) and by Buckwalter (37).

The effect of solar radiation is similar to that of heat. Wavelengths in the ultraviolet

up to 4000 Å are most effective in increasing the rate of oxidation (41, 43, 89, 287). With wavelengths of less than 2500 Å, photolysis, or the scission of chemical bonds, is the important reaction; but this reaction is inhibited by water and oxygen (148). This however, is not a factor with solar radiation which starts at 2900 Å (56). An important factor which increases the effect of light is photosensitization. Cellulose does not absorb radiations much above 2300 Å (43). Any dye or inorganic deposit which can absorb light and convey it to the reacting molecules increases the rate (69).

Treiber and Felbinger (273) claim that a small absorption band exists at 3500 Å. Kujirai (148) reports absorption bands at 1850 Å and 2537 Å. The function of weather protective treatments seems to be absorption of this radiation. Lead chromate which is effective absorbs strongly between 2200 and 5000 Å.

In this connection, an important survey of the literature on the action of light on textile materials was made by Appleby (9) and covers a period up to 1948.

There are other factors that pertain to the oxidation function during weathering. For example, free radical inhibitors increase the resistance to weathering (37). Metallic oxides which catalyze peroxide formation also catalyze the decomposition of cotton. Zinc oxide is an example of a metallic oxide which catalyzes the formation of hydrogen peroxide from water. Hydrogen peroxide has been proposed as the active compound by Baur and Neweiler (16) and by Egerton (69). Ozone accelerates the initiation of the oxidation of cellulose (27).

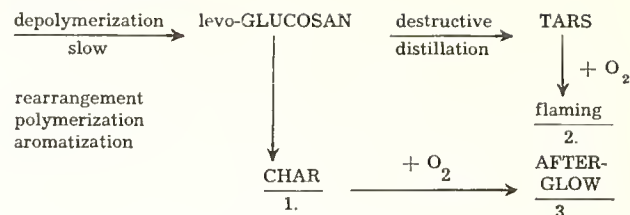
The action of atmospheric contaminants in the case of oxides of nitrogen is oxidation. This involves a hydrolytic decomposition which is dependent upon moisture content.

## FLAME RESISTANCE

The literature pertaining to the flammability of textile fibers was reviewed by Little (160), Gottlieb (103), McQuade (169), and others (45, 83, 131, 162, 201, 203, 219, 222, 232, 249).

The flammability of cellulose is due to the rapid oxidation of combustible tars formed at elevated temperatures. Cellulose, when heated above 275° F., decomposes as follows (109):

## CELLULOSE



The action of an ideal flameproofing compound or its decomposition products at flaming temperatures is threefold:

- The catalytic effect of the flameproofing compound increases char formation, and, therefore, decreases the fuel available for combustion. Theoretically, cellulose should decompose to carbon and water (160, 161). Many compounds act as Lewis acids and catalyze the dehydration of the cellulose (250). Basic flameproofing compounds catalyze rearrangements which also result in an increase of char formation (73).
- Lowering the combustibility of the tars (i. e., flameproofing the gas phase) is essential when the flameproofing compound used is an organic polymer, since its decomposition results in additional tars. For this purpose an atom or molecule with the ability to form relatively stable free radicals is incorporated in the polymer and acts as a chain breaker, inhibiting flaming (250).
- The inhibition of afterglow in the char is essential. Therefore, substances which promote the oxidation of the char to carbon monoxide are important. Because their reactions are weakly exothermic, the heat thereby produced is insufficient to propagate the afterglow. Silicon, boron, and phosphorus compounds are such effective substances inhibiting afterglow (160, 161, 201).

## WATER REPELLENCY

Keppler (140), Kogan (146), and Harding (116) have reviewed the literature on water repellency.

Treatments of fabrics for water repellency can be divided into two groups:

- Those in which the material is covered with a film or skin making it impermeable to air and moisture;

- b) Those in which the material is coated with a film having a high interfacial tension, thereby making the fibers hydrophobic.

The inorganic and organometallic compounds belong in this latter group. With the exception of organosilicon polymers, the durability of these finishes is limited.

Many inorganic and organometallic compounds have been found to impart water repellency to cotton fabrics.

### MISCELLANEOUS PROPERTIES

Modern chemical treatments have been discussed by Notarbartolo (197). Included in general reviews of finishes for cotton were those by Stott (263) by Little (160), by Krasny and Harris (147), by Goheen and DuPre' (99), and by Fisher (75).

Finishes that affect the physical properties

and appearance of cotton fabrics have been suggested. These properties include weighting, luster, shrink resistance, slip resistance, and stiffening.

Compounds that dissolve or partly dissolve the cellulose fibers generally also remove the fuzz fiber, partly swell the cellulose and bind the fiber, and tend to give increased luster, stiffness, and resistance to shrinkage and slip-page.

### LITERATURE FINDINGS CONCERNING CERTAIN COMPOUNDS APPLIED TO COTTON

The literature concerning specific compounds and the reported properties which they impart to cotton are given in a series of tables, as follows: Table I, Metallic Oxides; Table II, Inorganic Salts; Table III, Organometallic Salts and Chelates; and Table IV, Other Organometallic Compounds.



Table I.—Metallic oxides applied to cotton fabrics with properties reported

OXIDES OF—	APPLIED WITH—	PROPERTIES	COMMENTS	REFERENCES
ALUMINUM	Wax	Weighting and delustering Water repellent	Leaches; insoluble	4, 15, 77, 91, 116, 122, 206 207, 212, 213, 220, 237, 272
ANTIMONY	Titanium tetrachloride Chlorinated hydro- carbon Vinyl chloride Phosphates Urea formaldehyde	Weighting and delustering Flame resistant  Weather stable	Durable; insoluble	12, 13, 42, 80, 82, 101, 123, 134, 144, 160, 163, 164, 180, 201, 225, 238, 253, 269, 272, 275, 276, 277, 297, 298
ARSENIC	Chromium oxide	Mildew and rot resistant	Slightly soluble	23, 24
BISMUTH		Delustering and weighting	Insoluble	298
CERIUM		Mildew and rot resistant Weather stable	Insoluble	63, 77, 271
CHROMIUM	Urea formaldehyde Iron oxide Copper oxide	Weather stable Mildew and rot resistant	Durable; insoluble	18, 88, 128, 206, 240, 267, 272, 292
COPPER	Chromium oxide Chromium and iron oxides Iron oxide and wax	Mildew and rot resistant Weather stable Weather stable Water repellent	Leaches; insoluble Good durability	3, 18, 71, 74, 86, 128, 133, 204, 206, 234, 255, 292, 302
HAFNIUM		Weighting and delustering	Insoluble	288
IRON	Chlorinated paraffin Chromium and copper	Weighting and delustering Flame resistant	Durable; insoluble	18, 137, 193, 206, 243, 292
SILICON		Flame resistant		30, 49, 79, 131, 176, 222
THORIUM		Mildew and rot resistant Weather stable	Insoluble	210, 294
TIN	Chlorinated paraffin	Weighting and delustering Flame resistant	Insoluble	7, 206, 222, 243
TITANIUM	“Erfon” Antimony oxide ap- plied as chlorides Chlorinated paraffin Wax	Weighting and delustering Flame resistant and mildew and rot resistant Flame resistant Water resistant	Durable; insoluble	30, 61, 77, 107, 134, 150, 201 206, 226, 238, 252, 264, 272
TUNGSTEN		Flame resistant	Insoluble	45
ZINC	Chlorinated paraffin	Delustering and weighting Flame resistant Water repellent	Tenders fabric (slightly soluble)	44, 52, 77, 123, 127, 152, 157, 304
ZIRCONIUM	Wax	Weighting Water repellent Flame resistant	Insoluble	23, 24, 25, 26, 63, 66, 76, 116, 213, 237, 271, 282, 288

Table II.—Inorganic salts applied to cotton fabrics with properties reported

COMPOUND	APPLIED WITH—	PROPERTIES	SOLUBILITY <sup>1</sup>	REFERENCES
ALUMINUM chloride	Chlorinated paraffin	Flame retardant Water repellent; fire retardant; softening and sizing	Soluble Soluble hot water	47, 65, 95, 149, 198, 242 277, 278, 285
salts	Polyamines, formaldehyde, benzoic acid			
sulfate	Polyhydric alcohol		S., v. sl. s. alc.	
ANTIMONY borate oxychloride		Fire retardant Fire retardant	d. hot water; s. alc.	60, 106, 142, 185, 202, 203 265
phosphate		Fire retardant		
BARIUM salts sulfate		Water repellent Weighting	Insoluble	95, 210, 254
BORATES— BORIC ACID		Flame retardant	s.	1, 145, 192, 222
CADMIUM salts		Mildew and rot resistant		21, 187, 196, 294
CALCIUM carbonate	Antimony oxide Chlorinated paraffin	Fire retardant	Insoluble	121, 164, 198, 254
chloride sulfate		Deliquescent Weighting	Soluble Insoluble	
CERIUM salts	Aromatic polyamines formaldehyde, benzoic acid	Water repellent Mildew and rot resistant	i.; slowly oxidized in hot water; i. alc.; s. dil. a.	65, 210
CHROMIUM salts sulfate	Polphydric alcohol	Water repellent Fire retardant; softening; sizing	s.; i.a.	20, 36, 65, 149, 241, 246, 292
COPPER carbonate		Mildew and rot resistant Water repellent	i.; d. hot water	10, 18, 29, 50, 51, 57, 87,
cupramonium hydroxide	Aminated cellulose Urea formaldehyde resin	Mildew and rot resistant; improves luster; decreases friction	s.	90, 92, 102, 108, 128, 133,
phosphate		Water repellent	i.; sl. s. hot water	139, 186, 204, 239, 240, 241,
salts	Urea formaldehyde resin	Decreases friction		247, 258, 285, 300

See footnotes at end of table.

Table II.—Continued

COMPOUND	APPLIED WITH—	PROPERTIES	SOLUBILITY <sup>1</sup>	REFERENCES
IRON phosphate sulfate	Polyhydric alcohol	Water repellent Fire retardant; softening; sizing	i. s.; i. a.	20, 36, 202, 281, 285
LEAD chromate	Urea formaldehyde resin	Weather stable	i.	54, 58, 87, 88, 95, 96, 186,
salts	Aromatic polyamines	Weather stable Mildew and rot resistant Water repellency Drying		187, 221, 240, 251
MAGNESIUM ammonium phosphate chloride fluosilicate sulfate	Nitro-urea	Flame retardant	i.	19, 96, 199, 254
		Deliquescent Slip resistant Weighting	s. s. hot water s.	
MANGANESE salts	Urea formaldehyde resin	Driers Decreases friction	s.	19, 196, 210, 221
MONASTRAL BLUE	Fluosilicate Urea formaldehyde resin	Slip resistant Weather stable	i.	54
NICKEL salts		Water repellent		65, 200, 261
PHOSPHATES		Flame retardant	s.	1, 38, 145, 190, 192, 195, 199, 214, 275, 276
SIENNA	Urea formaldehyde resin	Weather stable	i.	54
SILICATE ammonium sodium		Flame resistant Slip resistant	s. sl. s.	11, 141, 175, 205, 214, 216, 244
SULFAMATES		Flame resistant	s.	218
THALLIUM carbonate salts		Mildew and rot resistant Water repellent	sl. s.	65, 128
TIN stannate phosphate		Improves luster Flame resistant	s. i.	159, 172, 173, 277

See footnotes at end of table.

Table II.—Continued

COMPOUND	APPLIED WITH—	PROPERTIES	SOLUBILITY <sup>1</sup>	REFERENCES
<b>TITANIUM</b>				
oxychloride salts		Flame resistant Flame resistant Water repellent Water repellent	d. hot water   i.	60, 65, 104, 201, 257, 261, 270, 286
sulfate				
<b>TUNGSTATE</b>				
sodium		Flame retardant	s.	222, 305
<b>ZINC</b>				
borate		Fire retardant		21, 36, 54, 67, 68, 82, 96,
carbonate		Flame retardant	i., s.a., s. alk.	108, 118, 119, 120, 121, 142,
chloride		Flame resistant; deliquescent	s.	143, 152, 153, 154, 155, 157,
chromate		Weather stable	0.1/100 ml.	172, 174, 175, 185, 187, 196,
salts	Urea formaldehyde	Weather stable		198, 203, 209, 242, 245, 261,
salts	Aminated cellulose	Weather stable; de- creases friction Mildew and rot resistant		307
sodium zincate		Improves luster		
<b>ZIRCONIUM</b>				
salts		Water repellent		65, 70, 261, 281, 291
ammonium dicarbonate- zirconylate		Water repellent		
<sup>1</sup>	S. sl. s. v. sl. s.	=soluble =slightly soluble =very slightly soluble	alc. i. dil.	=acid =dissolves =alkali



Table III.—*Organometallic salts and chelates applied to cotton fabrics with properties reported*

COMPOUND	APPLIED WITH—	PROPERTIES	COMMENTS	REFERENCES
<b>ALUMINUM SOAP</b>				
carboxymethyl cellulose		Mildew and rot resistant Water repellent	Most of these finishes are not durable to laundering even though they are insoluble in water	39, 46, 98, 176, 191, 198, 233, 235, 261, 266, 294
polyacrylate quinolinate tannate		Stable fiber  Weighting		
<b>BARIUM SOAP</b>				
		Water repellent		210, 294
<b>BERYLLIUM</b>				
carboxymethyl cellulose		Stable fiber		47
<b>CADMIUM SOAP</b>				
phenyl salicylate		Mildew and rot resistant		
penta chlorophenate	Urea formaldehyde resin	Water repellent  Mildew and rot resistant		21, 87, 176, 254, 294
<b>CALCIUM SOAP</b>				
carboxymethyl cellulose		Water repellent		
polyacrylate		Weighting Weighting		145, 198, 294
<b>COPPER SOAP</b>				
carboxymethyl cellulose		Mildew and rot resistant Water repellent		6, 10, 17, 18, 21, 51, 57, 74, 87, 94, 128, 129, 135,
glyoxime		Mildew and rot resistant		156, 158, 165, 176, 178, 179,
naphenate		Mildew and rot resistant		204, 208, 234, 235, 240, 246,
quinolinate		Water repellent		
		Mildew and rot resistant	Leaches	254, 267, 284, 293, 295, 296,
	Urea formaldehyde	Mildew and rot resistant	Durable	
phenyl salicylate		Mildew and rot resistant		301, 306
salt copolymer of maleic anhydride and styrene		Weighting Mildew and rot resistant		

Table III.—Continued

COMPOUND	APPLIED WITH—	PROPERTIES	COMMENTS	REFERENCES
CHROMIUM NAPHTHENATE		Mildew and rot resistant		176, 211
IRON tannate carboxymethyl cellulose		Mildew and rot resistant		235
LEAD carboxymethyl cellulose		Mildew and rot resistant		221, 235
MAGNESIUM tannate, quinolino- late		Mildew and rot resistant		176, 294
MERCURY SOAP carboxymethyl cellulose		Mildew and rot resistant Water repellent		18, 50, 76, 126, 136, 204, 235 295, 306
NICKEL carboxymethyl cellulose		Mildew and rot resistant		210, 235, 261, 306
TIN SOAP		Water repellent		261, 294
THORIUM SOAP		Water repellent		210, 261, 294
ZINC SOAP polyacrylate		Water repellent Weighting		198, 261, 294
ZIRCONIUM SOAP salt of co- polymer of maleic anhy- dride and styrene		Water repellent Weighting Mildew and rot resistant		70, 237, 261, 282, 283

Table IV.—*Other organometallic compounds applied to cotton fabrics with properties reported.*

METAL	COMPOUND	PROPERTIES	COMMENTS	REFERENCES
MERCURY	Phenyl mercury compounds	Mildew and rot resistant		6, 18, 50, 51, 62, 74, 76, 105, 126, 135, 204, 254, 256, 295
PHOSPHORUS	Brominated triallyl phosphate polymer	Flame resistant	Durable	7, 22, 38, 53, 64, 84, 85, 93,
	Bromoform triallyl phosphate polymer	Flame resistant	Durable	109, 110, 111, 112, 130, 131,
	Urea or melamine formaldehyde phosphate polymer	Flame resistant	Semidurable	167, 168, 171, 181, 182, 183,
	Tetrakis(hydroxymethyl)phosphonium chloride	Flame resistant	Durable	184, 189, 193, 194, 214, 215,
	Phosphonitrilic polymer	Mildew and rot resistant		227, 228, 229, 230, 231, 236,
		Flame resistant		290
SILICON	Siloxane	Water repellent	Durable	2, 14, 19, 31, 32, 33, 34, 35 49, 59, 71, 72, 79, 81, 97, 100, 116, 124, 132, 166, 188, 217, 242, 248, 261, 303, 308
TITANIUM	Ethyl orthotitanate	Water repellent		262
ZINC	Organo-zinc compounds	Mildew and rot resistant		18, 74, 108, 177
ZIRCONIUM	Ethyl zirconylate	Water repellent		170

# PART II—EXPLORATORY EVALUATION

by

**Pauline Beery Mack, Georgia Ellen Wright, and Ramon M. Esteve, Jr.**

## INTRODUCTION

## MATERIALS AND EXPERIMENTAL METHODS

Raw Cotton Used for Fabric Experiments

Fabric Specifications

Application of Finishes

Evaluation of Treated Fabrics

## RESULTS AND DISCUSSION

Retention of Add-on During Leaching

Retention of Add-on During Laundering

Strength Losses Through Application of Finish  
and During Storage

Abrasion Resistance

Mildew and Rot Resistance

Resistance to Outdoor Weathering and Actinic Degradation

Resistance to Heat Exposure

Flammability

Thermal Stability

Water Repellency

## CONCLUSIONS

# INTRODUCTION

Although a large number of investigations have been reported on the treatment of cotton with metallic compounds for the purpose of improving certain of its functional properties, no systematic screening of compounds through the Periodic Table of Elements has been reported, in which a battery of tests is employed in a single study. In numerous investigations, summarized in Part I of this report, many oxides and salts have been described as imparting such properties as improved mildew and rot resistance, resistance to weathering, water repellency, and flame resistance. Many compounds, however, have remained unexplored; and few of those studied have had more than a few properties evaluated in the same investigation.

This study included the screening of 74 oxides, hydroxides, and salts of metallic elements, involving 83 separate treatments, as possible finishes for cotton fabrics. Where other factors appeared equal, compounds producing white or cream-colored cotton fabrics were of greater preference because of their

greater range of end uses.

Several previously investigated compounds were included in this screening study for comparative purposes, because they appeared from the literature to have given the best results for certain purposes. These were: Aluminum stearate for its known property of water repellency; cupric oxide, cupric-8-quinolinolate, and two mercury compounds for the favorable results reported concerning the mildew and rot resistance which they impart to cotton; lead chromate for its demonstrated value in improving weather resistance (54, 251); and titanium dioxide and ferric hydroxide for their reported property of enhancing flame resistance in cotton fabrics.

The compounds used included 29 oxides and hydroxides, 14 phosphates, 11 sulfides, and 20 miscellaneous compounds. They were chosen so as to represent all groups in the Periodic Table of Elements where insoluble compounds are found, with emphasis on oxides and hydroxides.

## MATERIALS AND EXPERIMENTAL METHODS

### RAW COTTON USED FOR FABRIC EXPERIMENTS

Strict Middling cotton 1-1/16 inches, has a micronaire of 4.3, and Pressley Index of 78, scoured.

### FABRIC SPECIFICATIONS

The cotton fabrics used in this study were a 4-ounce, 38-in-wide muslin, 56 x 56 count, of 2-ply yarn made by the Philadelphia Textile Institute<sup>2</sup> to explicitly meet the specifications of the College of Household Arts and Sciences at the Texas Woman's University.

Type—American Upland

Grade—Strict Middling

Staple—1-1/8 inch

Preparation at Gin—Probably "B"

Area Grown—Mississippi Delta

Processed—Yarn combed and bleached

Twist/Inch—18.27 (single), 13.45 (ply)

Twist Direction—2 (single), S (ply)

Count—40.72/2

Tensile Strength—(120 yd. skein)

65 lbs. (singles), 129.2 lbs. (ply)

Single end of ply yarn has a tensile strength on the IP2 Tester of 480.4 grams. Elongation of ply yarn was 5.76%.

### APPLICATION OF FINISHES

Swatches 9 x 24 inches were used for the impregnations. After the fabrics were immersed in a bath they were passed through a squeeze roll, and allowed to air-dry. Immersion was repeated for a second bath; where fabrics were treated with sodium hydroxide a second bath was not used. Then they were rinsed, and air-dried again. All water used in the study was purified by the ion-exchange method.

Table V gives an outline of the solutions used in treating the cottons, together with any special methods of handling the applications.

<sup>2</sup> Mention of companies and trade products does not imply that they are recommended or endorsed by the U. S. Department of Agriculture over other companies and trade products not mentioned.



Table V.—Summary of treatment processes applied to cotton fabric, and responses of the treated cotton to leaching and laundering tests

Place of Major Element in Finishing Compound in Periodic Table	FINISHING COMPOUND <sup>1,2</sup>	TREATMENT BATHS		RESPONSE TO:	
		First Bath	Second Bath	Leaching	Laundering
ALUMINUM	Aluminum hydroxide (8.7) (light yellow)	20% aluminum acetate	15% sodium carbonate at 70° C. <sup>3</sup>	Satisfactory	Satisfactory
GROUP III A	Aluminum stearate (7.5) (white)	30 g. aluminum stearate dissolved in one liter of ligroin at 45° <sup>3</sup>		Good	Satisfactory
ANTIMONY	Antimonious oxide (3.0) (white)	50 g. of antimony trichloride in 500 ml. ethanol, 200 ml. water, and 50 ml. hydrochloric acid	15% sodium carbonate at 70° C. <sup>3</sup>	Unsatisfactory	Unsatisfactory
GROUP V A					
BARIUM	Barium chromate (10.0) (yellow)	5% barium chloride	10% sodium chromate at 70° C.	Satisfactory	Satisfactory
GROUP II A	Barium phosphate (7.6)	6% barium chloride	10% sodium phosphate adjusted to pH 7.0 with phosphoric acid, at 70° C.	Satisfactory	Satisfactory
	Barium sulfate (8.7) (white)	7% barium chloride	15% sodium sulfate at 70° C.	Good	Satisfactory
	Barium sulfate on mercerized cotton (10.8) (white)	Same	Same	Good	Satisfactory
BERYLLIUM	Beryllium oxide (1.9) (white)	7% beryllium nitrate	5% sodium hydroxide at 70° C.	Good	Satisfactory
GROUP II A					
BISMUTH	Bismuth hydroxide (7.8) (white)	45 g. of bismuth trichloride in 800 ml. of acetone	10% ammonium hydroxide at 70° C. <sup>3</sup>	Good	Satisfactory
GROUP V A	Bismuth phosphate (4.8) (white)	35 g. of bismuth trichloride dissolved in 300 ml. of water, 150 ml. ethanol, and 50 ml. hydrochloric acid	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Unsatisfactory
	Bismuth sulfide (5.1) (dark brown)	5% bismuth trichloride in 50-40-10 water, ethanol, hydrochloric acid	10% sodium sulfate at 70° C.	Good	Good
CADMIUM	Cadmium hydroxide (5.0) (white)	7% cadmium acetate	5% sodium hydroxide at 70° C.	Good	Good
GROUP II B					
CALCIUM	Calcium oxalate (9.5) (white)	7% calcium chloride	10% oxalic acid at 70° C.	Unsatisfactory	Unsatisfactory
GROUP II A	Cerous hydroxide (6.0) (cream)	7% cerous chloride	10% ammonium hydroxide at 70° C. <sup>3</sup>	Good	Good
CERIUM	Ceric oxide (5.0) (yellow)	7% ceric sulfate	8% sodium hydroxide at 70° C.	Satisfactory	Satisfactory
GROUP III B					
(Lanthanide Series)	Cerous phosphate (4.4) (tan)	7% cerous chloride	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Good

See footnotes at end of table.

Table V.—Continued

Place of Major Element in Finishing Compound in Periodic Table	FINISHING COMPOUND <sup>1,2</sup>	TREATMENT BATHS		RESPONSE TO:	
		First Bath	Second Bath	Leaching	Laundering
CHROMIUM GROUP VI B	Chromic oxide (1.8) (blue-gray)	6% chromium acetate This procedure was repeated twice	10% sodium carbonate at 70° C.	Satisfactory	Satisfactory
	Cobaltous chromate (3.5) (brown)	6% cobaltous acetate	10% sodium chromate at 70° C.	Satisfactory	Satisfactory
	Cobaltous hydroxide (4.5) (tan)	6% cobaltous acetate	5% sodium hydroxide at 70° C. Rinse in hot water	Satisfactory	Satisfactory
COBALT GROUP VIII	Cobaltous hydroxide (10.2) (tan)	20% cobaltous acetate	6% sodium hydroxide at 70° C.	Good	Good
	Cobaltous metaborate (6.0) (gray)	6% cobaltous acetate	10% sodium metaborate, with fabric kept in solution 20 minutes at 70° C.	Good	Good
	Cobaltous oxalate (5.5) (light pink)	Same	10% oxalic acid at 70° C.	Unsatisfactory	Unsatisfactory
COPPER GROUP I B	Cobaltous phosphate (4.7) (orchid)	6% cobaltous acetate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, but fabric was kept 20 minutes in this bath at 70° C.	Unsatisfactory	Unsatisfactory
	Cobaltous silicate (6.8) (tan)	6% cobaltous acetate	10% sodium silicate at 70° C.	Good	Good
	Cobaltous sulfide (5.3) (dark brown)	10% cobaltous acetate	10% sodium sulfide at 70° C.	Unsatisfactory	Unsatisfactory
COPPER GROUP I B	Cobaltous sulfite (1.8) (yellow-brown)	6% cobaltous acetate	10% sodium sulfite at 70° C.	Unsatisfactory	Unsatisfactory
	Cupric chromate (3.9) (yellow-green)	6% copper sulfate pentahydrate	10% sodium chromate, with fabric kept in solution 10 minutes at 70° C.	Satisfactory	Satisfactory
	Cupric oxalate (3.7) (light aqua)	6% copper sulfate pentahydrate	10% oxalic acid at 70° C.	Unsatisfactory	Unsatisfactory
COPPER GROUP I B	Cupric oxide (10.4) (green-brown)	12% copper sulfate	5% sodium hydroxide at 70° C.	Good	Satisfactory
	Cupric phosphate (7.2) (blue)	6% cupric phosphate pentahydrate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Good
	Cupric-8-quinolate (1.0) (green)	1% 8-hydroxy-quinoline in ethanol	1% copper sulfate at 70° C.	Good	Good
COPPER GROUP I B	Cupric sulfide (2.8) (greenish gray)	6% copper sulfate	10% sodium hydroxide at 70° C. <sup>3</sup>	Satisfactory	Satisfactory

See footnotes at end of table.



Table V.—Continued

Place of Major Element in Finishing Compound in Periodic Table	FINISHING COMPOUND <sup>1,2</sup>	TREATMENT BATHS		RESPONSE TO:	
		First Bath	Second Bath	Leaching	Laundering
IRON	Ferric hydroxide (5.1 (rust))	12% ferrous sul- fate heptahydrate	5% sodium hydroxide at 70° C.	Satisfactory	Satisfactory
	Ferric hydroxide (10.4) (rust)	12% ferric acetate and 3% hydrochloric acid	15% sodium carbonate at 70° C.	Good	Good
	Ferrous phosphate (3.0) (tan)	9% ferrous sulfate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Satisfactory
GROUP VIII	Ferrous sulfide (3.3) (orange)	8% ferrous sulfate heptahydrate	10% sodium sulfide at 70° C.	Satisfactory	Satisfactory
	Lanthanum oxide (8.8) (white)	8% lanthanum chloride	5% sodium hydroxide at 70° C. <sup>3</sup>	Good	Good
	Lead (plumbous) chromate (orange) (8.5)	9% lead acetate	10% sodium chromate at 70° C. <sup>3</sup>	Good	Satisfactory
LANTHANUM	Lead (plumbous) chromate on mercerized cotton (orange) (5.1)	7% lead acetate	10% sodium chromate at 70° C. <sup>3</sup>	Good	Satisfactory
	Lead (plumbous) hydroxide (white) (4.4)	7% lead acetate	10% ammonium hydrox- ide at 70° C. <sup>3</sup>	Satisfactory	Satisfactory
	Lead (plumbous) hydroxide (white) (8.4)	12% lead acetate	10% ammonium hydrox- ide at 70° C. <sup>3</sup>	Good	Good
GROUP IV A	Lead (plumbous) hydroxide (white) (15.3)	20% lead acetate	Same	Good	Good
	Lead (plumbous) metaborate (white) (6.9)	7% lead acetate	10% sodium meta- borate at 70° C.	Good	Good
	Lead (plumbous) molybdate (white) (10.2)	7% lead acetate	10% ammonium molybdate at 70° C.	Good	Good
MAGNESIUM	Lead (plumbous) oxalate (white) (7.8)	6% lead acetate	10% oxalic acid at 70° C.	Good	Good
	Lead (plumbous) phosphate (white) (8.7)	7% lead acetate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Good
	Lead (plumbous) silicate (white) (3.6)	7% lead acetate	11% sodium silicate at 70° C.	Unsatisfactory	Satisfactory
GROUP II A	Lead (plumbous) sulfide (dark brown) (7.9)	7% lead acetate	10% sodium sulfide at 70° C.	Good	Good
	Magnesium fluoride 10.2) (white)	20% magnesium sul- fate duodecahydrate	10% sodium fluoride	Good	Good
	Magnesium oxide (7.9) (white)	20% magnesium sul- fate heptahydrate	5% sodium hydroxide at 70° C. <sup>3</sup>	Unsatisfactory	Unsatisfactory
GROUP II A	Magnesium oxide (10.5) (white)	30% magnesium sul- fate heptahydrate	Same <sup>3</sup>	Unsatisfactory	Unsatisfactory
	Magnesium oxide (13.5) (white)	40% magnesium sul- fate heptahydrate	6% sodium hydroxide at 70° C. <sup>3</sup>	Unsatisfactory	Unsatisfactory
	Magnesium phosphate (7.6) (white)	20% magnesium sul- fate duodecahydrate	10% sodium phosphate adjusted to pH 7.0 with phosphoric acid, at 70° C.	Unsatisfactory	Unsatisfactory

See footnotes at end of table.

Table V.—Continued

Place of Major Element in Finishing Compound in Periodic Table	FINISHING COMPOUND 1,2	TREATMENT BATHS		RESPONSE TO:	
		First Bath	Second Bath	Leaching	Laundering
MANGANESE  GROUP VII B	Manganous oxide (14.5) (red-brown)	12% manganous chloride	5% sodium hydroxide at 70° C.	Good	Satisfactory
	Manganous phosphate (4.0) (white)	8% manganous chloride	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Good
	Manganous silicate (7.0) (pale tan)	8% manganous chloride	10% sodium silicate, with pH adjusted to pH 12 with phosphoric acid, at 70° C.	Good	Good
	Manganous sulfide (7.1) (brown)	6% manganous chloride	10% sodium sulfide at 70° C.	Good	Satisfactory
MERCURY	Mercuric oxide (0.7) (greenish-yellow)	8% mercuric oxide in 75-25 water-HCl	5% sodium hydroxide at 70° C.	Unsatisfactory	Unsatisfactory
GROUP II B	Mercurous chloride (7.5) (white)	5% mercurous nitrate	10% sodium chloride at 70° C.	Unsatisfactory	Unsatisfactory
MOLYBDENUM  GROUP VI B	Molybdenum oxide (2.8) (gray)	Molybdenum metal was dissolved in a mixture of hydrochloric and nitric acids, which was added slowly. The reaction was exothermic. The solution had an orange color. Additional molybdenum metal was added, after which a further reaction took place resulting in a blue-green solution because of the formation of a molybdenum oxy-chloride. This compound was dissolved in ethyl alcohol, filtered, and applied to the fabric. The oxide was formed by immersing the fabric in 5 percent sodium hydroxide. The different levels of add-on represented slightly different lengths of time in the solutions.			
	Molybdenum oxide (4.1) (gray)				
NICKEL	Nickelous oxide (3.7) (light brown)	7% nickelous chloride	5% sodium hydroxide at 70° C.	Unsatisfactory	Unsatisfactory
GROUP VIII	Nickelous phosphate (5.1) (cream)	6% nickelous chloride	10% sodium phosphate, pH adjusted to 7.0 with phosphoric acid, at 70° C.	Satisfactory	Satisfactory
SILICON  GROUP IV A	Nickelous sulfide (3.5) (dark gray)	6% nickelous chloride	10% sodium sulfide at 70° C.	Unsatisfactory	Unsatisfactory
	Silicon dioxide (3.0) (white)	The fabric was padded to a 150 percent pick-up in a solution of 30 g. tetrakis (2-methoxyethyl) silicate dissolved in 450 ml. of ethanol. After rinsing and drying the fabric was cured for 30 minutes at 120° C.			
SILVER  GROUP I B	Silver oxide (13.8) (dark brown)	12% silver nitrate	5% sodium hydroxide at 70° C.	Good	Good
	Silver phosphate (12.5) (gray)	6% silver nitrate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Good	Good
	Silver sulfide (5.5) (dark brown)	5% silver nitrate	8% sodium sulfide at 70° C.	Good	Good

See footnotes at end of table.

Table V.—Continued

Place of Major Element in Finishing Compound in Periodic Table	FINISHING COMPOUND <sup>1,2</sup>	TREATMENT BATHS		RESPONSE TO:	
		First Bath	Second Bath	Leaching	Laundering
TELLURIUM	Tellurium oxide (0.5) (white)	2 paddings with asaturated solution of tellurium chloride in hydrochloric acid	10% ammonium hydroxide at 70° C.	Satisfactory	Satisfactory
GROUP III A					
THORIUM	Thorium hydroxide (7.1) (white)	10% thorium chloride	5% sodium hydroxide at 70° C.	Good	Good
GROUP III B					
(Actinide Series)	Thorium sulfide 6.5) (light gray)	10% thorium chloride	10% sodium sulfide at 70° C.	Good	Good
TIN	Tin (stannous) hydroxide (white) (8.4)	7% stannous chloride	5% sodium hydroxide at 70° C.		
GROUP IV A					
	Tin (stannic) oxide (4.9)	8% stannic chloride	15% sodium carbonate at 70° C. <sup>3</sup>	Good	Good
			10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Satisfactory	Satisfactory
	Tin (stannous) phosphate (off-white) (7.2)	6% stannous phosphate			
	Tin (stannic) sulfide (cream) 1.5)	6% stannic chloride	10% sodium sulfide at 70° C.	Satisfactory	Satisfactory
TITANIUM	Titanium dioxide (0.1) (white)	11% titanium tetra-chloride in 70-30 water-alcohol with 5% hydrochloric acid	15% sodium carbonate at 70° C. <sup>3</sup>	Good	Good
GROUP IV B					
TUNGSTEN	Tungsten oxide (2.5) (cream)	8% sodium tungstate	10% hydrochloric acid at 70° C. <sup>3</sup>	Satisfactory	Unsatisfactory
GROUP VI B					
URANIUM	Uranyl phosphate (6.5) (light yellow)	6% uranyl nitrate hexahydrate	10% sodium phosphate, adjusted to pH 7.0 with phosphoric acid, at 70° C.	Unsatisfactory	Unsatisfactory
(Actinide Series)					
VANADIUM	Vanadium oxide (2.0) (tan)	2 paddings with a saturated solution of divanadyl tetra-chloride in hot ethyl alcohol	5% sodium hydroxide at 70° C.	Unsatisfactory	Unsatisfactory
GROUP V B					
ZINC	Zinc oxide (9.0) (white)	Zinc oxide was prepared by precipitating 80 g. of zinc chloride with ammonium hydroxide. An excess of ammonium hydroxide was added to dissolve the hydroxide. The fabric was padded with this solution to 16.0 percent and was placed outside to dry. It then was rinsed twice, allowed to dry, cured at 120° C. for 30 minutes, and again rinsed thoroughly.		Satisfactory	Satisfactory
GROUP II B					
	Zinc phosphate (14.8) (white)	11% zinc chloride	10% sodium phosphate, adjusted to pH 8.0 with phosphoric acid	Good	Good
	Zinc sulfide (6.5) (light cream)	5% zinc chloride	10% sodium sulfide at 70° C.	Good	Satisfactory
ZIRCONIUM	Zirconium hydroxide (white) (9.2)	6% zirconyl nitrate	15% sodium carbonate at 70° C.	Good	Good
GROUP IV B					

<sup>1</sup> The number in parentheses after the name of the compound refers to the percent add-on.<sup>2</sup> The designation in parentheses under the name of the compound refers to the resulting color of the treated cotton.<sup>3</sup> The treated cotton was cured for 20 minutes at 120° C.



## EVALUATION OF TREATED FABRICS

The major test methods used were those of the American Association of Textile Chemists and Colorists (5), the American Society for Testing Materials (8), the U. S. Federal Supply Service Federal Specifications (279), and the National Bureau of Standards Commercial Standards (280). Tests not derived from one or more of these sources are described.

The untreated control fabric and the treated cotton fabrics were evaluated by the following tests:

- 1) **Resistance to Leaching** (279-f).
- 2) **Resistance to Laundering.** The test fabrics were laundered by the ASTM and Commercial Standard techniques (8-b, 280-b). The extent to which the add-on had been lost by leaching or by laundering was determined by ashing specimens of the treated samples and finding the differences in weight. Because of the loosely held surface deposit, the loss of compound tended to be greater during the first leaching or laundering than during subsequent tests.

In evaluating the loss of add-on during leaching or laundering, arbitrary ranges were adopted to rate the permanence of the treatment. After 2 successive leachings retention of 80 percent or more of the compound was regarded as **good**, retention of 79 to 60 percent as **satisfactory**, and retention below 60 percent as **unsatisfactory**.

- 3) **Breaking Strength, Standard Conditions** (8-a, 279-a). **Breaking Strength, Wet.** Specimens wet according to a Commercial Standard method (280-a) and tested according to ASTM and Federal Specifications (8-a, 279-a).
- 4) **Bursting Strength, Standard Conditions** (8-a, 279-b, 280-a). **Bursting Strength, Wet.** Specimens wet according to a Commercial Standard method (280-a) and tested according to ASTM and Federal Specifications (8-a, 279-b).
- 5) **Tearing Strength, Standard Conditions** (279-c). **Tearing Strength, Wet.** Specimens wet according to a Commercial Standard method (280-a) and tested according to Federal Specifications (279-c).

In making evaluations to determine excessive strength losses as a result of either the application treatment or of darkroom storage, all types of strength measurements were taken into consideration.

Treatments were considered **satisfactory** where fabric strength losses after treatment were less than 15 percent. Losses of 15 percent or more were regarded as **unsatisfactory**. Particular attention was paid to the corresponding strength losses at standard conditions and in the wet state. When the wet strength losses exceeded those in the conditioned state, it was concluded that damage to the cellulose complex may have taken place. In the few cases where this situation occurred, the strength losses markedly exceeded 15 percent.

In some cases decreases in tear strength were observed without a corresponding decrease in breaking strength. A situation of this type would tend to indicate crosslinking between the cellulose and the finishing compound.

After 6 months of darkroom storage, total losses in strength no greater than 20 percent were regarded as **satisfactory**.

- 6) **Abrasion Resistance.** Taber Abraser<sup>2</sup> rotary platform method (279-g), as modified by Zook (309), following the mounting method of Zook (310) and of Thomas and Wham (268).
- 7) **Mildew and Rot Resistance.** Soil burial method (8-e, 279-e). Although the temperature and humidity were controlled throughout this test, the results of the soil burial test should not be regarded as strictly quantitative. In some cases the determination followed kinetics of the zero order. In other samples, a sharp change in rate was observed during the test. The time in days required for a textile specimen to lose one-half of its wet tensile strength was used as the basis for computing the results of this test.
- 8) **Resistance to Outdoor Weathering.** Frames were constructed similar to those used at the Southern Regional Research Laboratory for the same type of test (58, 279-i). The specimens, 9 x 18 inches warpwise, were placed on the inclined rack at a 45° angle facing south.

The resistance to deterioration was measured from the loss in breaking strength. The loss of strength resulting from weathering was observed to follow the kinetics of a first order process in the limits imposed by the variations in weathering conditions inherent in this test. The rates were calculated from the following equation:

$$\log \frac{T.S._o}{T.S._t} = kt$$

where

T.S.<sub>o</sub> is the initial strength of the fabric (pounds);

T.S.<sub>t</sub> is the strength after time t, (pounds); t is the time of exposure (months).

Relative stability to weathering was based on breaking-strength losses of the treated as compared with the untreated fabric, i.e.

$$\text{Relative Stability} = \frac{\text{Rate for treated fabric}}{\text{Rate for untreated control}}$$

- 9) **Resistance to Actinic (Fade-Ometer 2/) Degradation (5-a).** Strips raveled to 1-inch width, as for breaking-strength evaluation, were placed in the specimen holders (in a horizontal position) of the FDA-R Type Fade-Ometer. These holders were placed on the rotating racks and were rotated at 53° C. for 100-, 200-, and 400-hour periods, respectively.

At the end of the respective exposure times, one-half of the strips were subjected to breaking-strength tests in the conditioned state and one-half in the wet state.

The reported data are based on the 400-hour exposure time.

- 10) **Resistance to Heat Degradation (279-h).** The test was made in an oven at 140° C.

with test strips 6 inches long raveled to a 1-inch width, as for breaking-strength tests. The test was continued for 120 hours.

The rates of degradation from exposure to heat at the designated oven temperature were calculated on the basis of breaking-strength loss. The rate of strength loss followed the kinetics of a first order process. The results were calculated and reported as in the case of outdoor weathering.

- 11) **Flammability, 45° Angle Test (5-b, 8-f). Flammability Verticle Flame Test (8-f).**

Treated fabrics which did not pass the 45° angle test were recorded as **failed**. Those which did not pass this test but for which the rate of burning was retarded were recorded as **failed, slower burning**. All treated cottons were given the 45° angle test; but only the fabrics passing this test were subjected to the vertical flame test.

- 12) **Thermal Stability** was determined from the rates of pyrolytic decomposition of the treated and untreated cotton fabrics at 310° C. The rates were determined from the first order logarithmic plot described by the following equation:

$$\log \frac{W_t - W}{W_o - W} = kt$$

where

W<sub>o</sub> is the initial weight of the fabric (grams);

W<sub>t</sub> is the weight of char after pyrolysis for time t; W is the weight of char after pyrolysis for 10 times the half life of the pyrolytic degradation; and t is the time of pyrolysis (minutes).

The results are reported as relative stability, i.e., rate of decomposition of treated fabric divided by rate of the control.

- 13) **Water Repellency — Spray Test (8-c, 279-d).**

## RESULTS AND DISCUSSION

The colors of the treated fabrics ranged from white to dark brown. Although the darker colors would limit the end uses of cotton fabric chiefly to agricultural purposes, the perform-

ance of some of these would recommend further consideration of their possible applications for certain purposes.

Tables V, VI, and VII summarize the findings of the study.

Table V lists the finishing compounds alphabetically, and the following data are recorded for each compound: Position of major element of the finishing compound in the Periodic Table, percent add-on, treatment baths, resulting color of treated fabric, and response to leaching and launderings.

Table VI gives the findings on all the treated cotton studied in the screened tests. The results are given by numerical value, in most

cases relative to the behavior of the untreated fabric. The results tabulated in this table relate to mildew and rot resistance, weathering, actinic Fade-Ometer degradation, flammability, thermal stability, aging in the oven, and water repellency.

Table VII lists the Light Screening Index of 59 inorganic compounds on cotton fabrics as obtained from the light absorption by the Du-Spectrophotometer<sup>2</sup>, and the relative rates of aging at 140° C in comparison with the relative rate of weathering.



Table VI.—Results of the Evaluation test of treated cotton fabrics

Sample No.	Finishing Compound	Add-on (%)	Resistance to Mildew and Rot (Days)	Weathering Stability (Relative)	Fade-Ometer at 53° C. (Relative)	Flammability 45° Vertical (Seconds)	Thermal Stability (Relative)	Ageing at 140° C. (Relative)	Water Repellency (Spray Rating)
—	Control	0.0	2	1.0	1.0	13 A. G. <sup>1</sup>	1.0	1.0	0
34	Aluminum hydroxide	3.7	4	1.7	1.6	A. G.	0.3+	1.0	0
1	Aluminum stearate	7.5	2	1.0	0.7	F. S. 1" B. C. <sup>3</sup>	...	0.6	0
32	Antimonious oxide	3.0	2+	0.8	1.0	20 A. G.	0.9	1.0	0
21	Barium chromate	10.0	1+	1.3	2.3	21 A. G.	1.5	1.0	50
69	Barium phosphate	7.6	1+	0.7	1.0	32 A. G.	0.8+	1.0	0
6	Barium sulfate	8.7	2	1.1	0.7	17 A. G.	1.0	1.0	0
	Barium sulfate on mercerized cotton	10.8							
65	Beryllium oxide	1.9	5	0.3	0.9	38 A. G.	0.2—	0.6	0
27	Bismuth hydroxide	7.8	3+	0.1	0.6	13 A. G.	0.1	1.0	50-70
88	Bismuth phosphate	4.8	1+	1.0	1.3	14 A. G.	0.4	0.8	0
89	Bismuth sulfide	5.1	1+	1.0	0.9	18 A. G.	0.6	1.0	0-50
30	Cadmium hydroxide	5.0	2	0.8	0.9	44 Char	0.9	1.1	0
101	Calcium oxalate	9.5	1+	1.3		22 A. G.	0.8		0
24	Cerous hydroxide	6.0	4	1.4	1.3	19 A. G.	1.0	1.6	50
54	Ceric oxide	5.0	2	0.6	1.0	A. G.	0.8+	0.5	0
72	Cerous phosphate	4.4	2	0.5	0.1	F. S. 1" B. C.	0.7+	0.5	0
26	Chromic oxide	1.8	2+	0.9	1.4	19 A. G.	0.7+	0.9	0
						A. G.			
						F. S. 1" B. C.			
41	Cobaltous chromate	3.5	13	1.7	2.1	16 A. G.	1.0	0.8	80
29	Cobaltous hydroxide	4.0	12	5.4	2.5	13 A. G.		1.1	0
31	Cobaltous hydroxide	5.6	10	6.0		13 A. G.	0.7		0
44	Cobaltous metaborate	6.0	16	2.3	3.3	15 A. G.	0.9	0.9	70
102	Cobaltous oxalate	5.5	1+	1.1		13 A. G.	1.0		0
43	Cobaltous phosphate	4.7	4+	1.1	0.7	15 A. G.	0.3	0.6	0
45	Cobaltous silicate	6.8	11+	1.7	2.2	22 A. G.	1.0	0.5	0
91	Cobaltous sulfide	5.3	8	2.5	0.5	10 A. G.	0.1+	1.3	0
42	Cobaltous sulfite	1.8	7	1.6	1.5	14 A. G.	0.4	0.9	70-80
46	Cupric chromate	3.9	100	0.7	0.7	16 A. G.	1.0	0.5	0
103	Cupric oxalate	3.7	14	0.5		16 A. G.	1.0		0
39	Cupric oxide	10.4	100	1.6	0.9	16 A. G.	1.2	0.5	50
64	Cupric phosphate	7.2	100	1.5	0.7	37 A. G.	0.2—	1.1	0
5	Cupric-8-quinolinolate	1.0	6	0.8	0.9	18 A. G.		0.4	0
50	Cupric sulfide	2.8	100	0.5	1.8	26 A. G.	0.2+	0.5	50
28	Ferric hydroxide	5.1	2+	0.9	0.9	A. G.	0.7+	0.6	0
						F. S. 1" B. C.			
40	Ferric hydroxide	10.4	5	0.4	0.6	A. G.	0.6—	0.4	0-50
						F. S. 1" C. L. 4" <sup>4</sup>			
67	Ferrous phosphate	3.0	1+	0.7	0.7	32 A. G.	0.7—	0.3	0
53	Ferrous sulfide	3.3	2	0.5	0.7	A. G.	0.5+	0.7	0
75	Lanthanum oxide	8.8	1+	1.5	0.1	F. S. 1" B. C.	1.1	0.8	0
8	Lead (plumbous) chromate	8.5	2	6.7	1.9	13 A. G.	2.0	1.5	0
						A. G.			
						F. S. 3" B. C.			
	Lead (plumbous) chromate on Mercerized cotton	5.1							
33	Lead (plumbous) hydroxide	4.4	1+	0.9	0.6	A. G.	1.5	0.8	70-80
112	Lead (plumbous) hydroxide	8.4				F. S. 1" C. L. 9" C. L. 7"			0-50

See footnotes at end of table.



Table VI.—Continued.

Sample No.	Finishing Compound	Add-on (%)	Resistance to Mildew and Rot (Days)	Weathering Stability (Relative)	Fade-Ometer at 53° C. (Relative)	Flammability 45° Vertical (Seconds)	Thermal Stability (Relative)	Ageing at 140° C. (Relative)	Water Repellency (Spray Rating)
111	Lead (plumbous) hydroxide	15.3				C. L. 6"	1.1+		50-70
57	Lead (plumbous) metaborate	6.9	2	0.9	0.4	A. G. F. S. 1" B. C.	1.2	0.1	0
56	Lead (plumbous) molybdate	10.2	2+	0.3	0.6	35 A. G.	0.3—	0.3	0
104	Lead (plumbous) oxalate	7.8	3	1.1		18 A. G.	1.3		0
22	Lead (plumbous) phosphate	8.7	2	0.8	1.0	14 A. G.	0.3	0.7	0
23	Lead (plumbous) silicate	3.6	2	1.1	0.9	A. G. F. S. 1" B. C.	1.4	1.0	0
48	Lead (plumbous) sulfide	7.9	4	0.6	1.0	12 A. G.	1.3+	1.0	50
84	Magnesium fluoride	10.2	1+	0.9	0.8	25 A. G.	0.7—	0.9	0
106	Magnesium oxide	7.9	1.0+			30 A. G.			0
37	Magnesium oxide	10.5	1.0	2.3	1.0	60 A. G.	1.2	1.2	0
105	Magnesium oxide	13.5	1.0			F. S. 1" B. C.			0
86	Magnesium phosphate	7.6	1+	1.8	0.9	25 A. G.	0.3+	0.8	0
38	Manganous oxide	14.5	1+	2.1	2.2	A. G. F. S. 1" B. C.	1.2	0.6	0-50
83	Manganous phosphate	4.0	1+	2.0	1.9	15 A. G.	0.8	1.3	0
82	Manganous silicate	7.0	1+	1.6	1.7	50 A. G.	1.3	1.0	0
49	Manganous sulfide	7.1	2	0.7	1.1	40 A. G.	1.0	0.5	0
58	Mercuric oxide	0.7	100	0.6	0.9	16 A. G.	1.0—	0.5	0
93	Mercurous chloride	7.5	100	0.1	0.3	10 A. G.	0.4	0.9	0
66	Molybdenum oxide	2.8	1+	0.3	1.1	A. G. F. S. 1" B. C.	0.2	1.0	0
55	Molybdenum oxide	4.1		1.0	0.5			0.5	0
61	Nickelous oxide	3.7	6	1.3	0.4	14 A. G.	0.8+	0.3	0
70	Nickelous phosphate	5.1	4	1.6	1.4	20 A. G.	0.3+	0.6	50
81	Nickelous sulfide	3.5	10	1.7	0.3	20 A. G.	0.1+	0.2	50
78	Silicon dioxide	3.0	+	0.7	1.1	16 A. G.	0.7—	1.2	0
63	Silver oxide	13.8	2+	0.3	0.7	17 A. G.	0.4	0.5	0
68	Silver phosphate	12.5	8	1.0	0.8	32 Heavy Char	0.1+	0.4	0
92	Silver sulfide	5.5	1+	1.6	2.5	10 A. G.	0.3	0.3	0
73	Tellurium oxide	0.5	1+	3.0	0.8	13 A. G.	1.0—	0.3	0
25	Thorium hydroxide	7.1	4+	0.5	1.0	15 A. G.	0.4	0.7	0
90	Thorium sulfide	6.5	2+	1.1	1.6	12 A. G.	0.8	0.2	0-50
62	Tin (stannous) hydroxide	8.4	2+	0.2	1.1	A. G. F. S. 1" B. C.	0.2—	0.4	0
35	Tin (stannic) oxide	4.9	3	0.3	0.7	A. G. F. S. 1" B. C.	0.4	0.7	0
71	Tin (stannous) phosphate	7.2	2	0.9	0.9	41 A. G.	0.4—	0.3	0
51	Tin (stannic) sulfide	1.5	2	0.3	0.3	9 A. G.	0.5+	0.1	0
16	Titanium dioxide	6.1	5	0.2	0.6	Did not ignite	0.4	0.6	0
87	Tungsten oxide	2.5	2	0.6	0.1	A. G. C. L. 8"	0.2	1.0	0
79	Uranyl phosphate	6.5	1+	0.3	0.8	10 A. G.	0.8	1.0	0
60	Vanadium oxide	2.0	2	0.1	0.9	40 A. G. 25 A. G.	0.7+	0.5	0
47	Zinc oxide	9.0	5+	0.6	1.0	34 A. G.	0.8—	0.6	0-50
94	Zinc phosphate	14.8	2+	1.1	0.9	15 A. G.	0.3+	1.8	0
52	Zinc sulfide	6.5	2+	0.1	0.7	45 A. G.	1.0+	0.8	50
36	Zirconium hydroxide	9.2	3	1.2	1.3	46 A. G.	0.7+	0.9	0

<sup>1</sup> Afterglow<sup>2</sup> Flame stopped<sup>3</sup> Burned completely.<sup>4</sup> Char light.

Table VII.—*Light screening index of 59 inorganic compounds on cotton fabrics as obtained from the light absorption by the Du-Spectrophotometer and the relative rates of aging at 140° C. in comparison with the relative rate of weathering*

Sample No.	Compound Used As Finish	RELATIVE RATE				
		Add-on (%)	Weathering	Light Absorption (I)	Aging 140° C. (C)	Light Index (IC)
Control	Untreated	0.0	1.0	1.0	1.0	1.0
34	Aluminum hydroxide	3.6	1.7	1.0+	0.7	0.7
1	Aluminum stearate	7.5	1.0	1.1	0.6	0.7
13	Aluminum stearate	7.5	1.0	1.4	0.6	0.8+
32	Antimonious oxide	3.0	0.8	1.3	1.0	1.3
21	Barium chromate	10.0	1.3	3.6	1.0	3.6
69	Barium phosphate	7.6	0.7	1.0	0.6	0.6
6	Barium sulfate	8.7	1.1	1.0	1.0	1.0
65	Beryllium oxide	1.9	0.3	1.1	1.0	1.1
27	Bismuth hydroxide	7.8	...	2.0	0.6	1.2
30	Cadmium hydroxide	5.0	0.8	1.0	1.1	1.1
24	Cerous hydroxide	6.0	1.4	2.0+	1.6	3.2
54	Ceric oxide	5.1	0.6—	3.0	0.5—	1.5—
72	Cerous phosphate	4.4	0.5	1.4—	0.4	0.6
26	Chromic oxide	1.8	0.9	1.3	0.9	1.1
41	Cobaltous chromate	3.5	1.7	...	1.0	...
29	Cobaltous hydroxide	4.0	5.4	2.6, 6.7	1.1	6.0
31	Cobaltous hydroxide	5.6	5.3	...	1.2	...
44	Cobaltous metaborate	6.0	2.3	2.0	1.0	2.0
43	Cobaltous phosphate	4.7	1.1	1.1	0.7	0.8
45	Cobaltous silicate	6.8	1.7	1.7+	0.5	0.8
42	Cobaltous sulfite	1.8	1.6	4.0	1.0	4.0
46	Cupric chromate	3.9	0.7	5.0	0.5	2.5
39	Cupric oxide	10.4	1.6	4.0	0.5	2.0
64	Cupric phosphate	7.2	1.5	2.0	0.6	7.2
5	Cupric-8-quinolinolate	1.0	0.8	2.9	0.4	1.1
50	Cupric sulfide	2.8	0.5	5.0	0.1	0.5
28	Ferric hydroxide	5.1	0.9	5.0+	0.6	3.0
40	Ferric hydroxide	10.4	0.4	5.0+	0.4	2.0
67	Ferrous phosphate	3.0	0.7—	3.0	0.4—	1.2
53	Ferrous sulfide	3.3	0.5	5.0	0.5—	2.5
75	Lanthanum oxide	8.8	1.5	1.0	1.0	1.0
8	Lead (plumbous) chromate	8.5	6.7	4.0	1.5	6.0
33	Lead (plumbous) hydroxide	4.4	0.9	1.1	0.8	0.9
57	Lead (plumbous) metaborate	6.9	0.9	1.0	0.5—	0.5
56	Lead (plumbous) molybdate	10.2	0.3	3.0	0.1+	0.3
22	Lead (plumbous) phosphate	8.7	0.8	1.1	0.7	0.7
23	Lead (plumbous) silicate	3.6	...	1.0	...	...
48	Lead (plumbous) sulfide	7.9	0.6	7.0	2.6	18.0+
37	Magnesium oxide	10.5	2.3	1.1	1.2	1.3
38	Manganous oxide	14.5	2.1	10.0	0.6	6.0+
49	Manganous sulfide	7.1	0.7	10.0	0.5	5.0+
58	Mercuric oxide	0.7	0.6	4.5	0.5+	2.0
66	Molybdenum oxide	2.8	0.3	3.1	0.3+	0.9
55	Molybdenum oxide	4.1	1.0	2.3	0.3	0.7
61	Nickelous oxide	3.7	1.3—	2.4	0.4	1.0
70	Nickelous phosphate	5.1	1.6	1.0	0.3—	0.3—
63	Silver oxide	13.8	0.3	10.0	1.1	11.0
68	Silver phosphate	12.5	1.0	4.3	1.0	4.3
73	Tellurium oxide	0.5	3.0	1.3	0.9	1.2
25	Thorium hydroxide	7.1	0.5	1.1+	0.7	0.8
62	Tin (stannous) hydroxide	8.4	0.2+	3.6	0.5+	1.8
35	Tin (stannic) oxide	4.9	0.3	1.0+	0.7	0.7
71	Tin (stannous) phosphate	7.2	0.9	1.0	0.5—	0.5
51	Tin (stannic) sulfide	1.5	0.3	1.1+	0.8	0.9
16	Titanium dioxide	6.1	0.2	2.0	0.6	1.2
60	Vanadium oxide	2.0	0.1	4.0	0.3—	1.2
47	Zinc oxide	9.0	0.6+	2.0—	0.4	0.8—
52	Zinc sulfide	6.5	0.1	2.0	0.7	1.4
36	Zirconium hydroxide	9.2	1.2	1.2	0.9	1.1

## RETENTION OF ADD-ON DURING LEACHING

Eighteen compounds were classed as **unsatisfactory** on the basis of the leaching test, which involved two successive treatments (Table V). Although the complete battery of tests was given to the fabrics finished with the compounds in Table V, those failing the leaching test were considered as unsuitable for further study (see Part III).

Of the compounds rated as unsatisfactory after leaching, none added any highly desirable properties to the cotton except the two mercury compounds and magnesium oxide. Both mercuric oxide and mercurous chloride gave very good results in the test for relative stability to mildew and rot. Magnesium oxide treatment was rated good after outdoor weathering exposure.

## RETENTION OF ADD-ON DURING LAUNDERING

Nineteen compounds were rated **unsatisfactory** in the laundering test. Except for three compounds these corresponded with the leaching test. Bismuth phosphate and tungsten oxide treated cotton, were rated as unsatisfactory after laundering but not to leaching. Lead silicate treatment was satisfactory after the laundering test, but unsatisfactory to leaching.

Compared to the leaching test, the more rigorous laundry test placed a larger number of treatments in the satisfactory and a smaller number in the good classification. Two additional treatments were eliminated in the laundry test (bismuth phosphate and tungsten oxide) were not found to add any particularly desirable characteristics to cotton fabric within the scope of the tests applied in this study.

## STRENGTH LOSSES THROUGH APPLICATION OF FINISH AND DURING STORAGE

Only six types of treated fabric displayed **unsatisfactory** strength losses, within the definition of the term, as a result of the application of the finish. These treatments included: Vanadium oxide, molybdenum oxide (2.8, 4.1 percent add-on), tungsten oxide, silver oxide, tellurium oxide, and tin (stannous) phosphate. Three finishes were early known to be unsuitable because of unsatisfactory response to the

leaching or to the laundering test. It was found, however, that none of these finishes added any noteworthy properties to cotton.

Only two treatments caused a strength loss during darkroom storage sufficiently great to place them in the **unsatisfactory** category as defined for purposes of this report. These were cobaltous sulfite, eliminated because of unsatisfactory response to the leaching and laundering tests, and cobaltous chromate. The latter gave **good** results both in the soil burial test and the test for relative stability to actinic degradation.

None of the treated fabrics exhibited proportionately higher tear than breaking strength losses. As noted, this would be regarded as denoting probable crosslinkage.

## ABRASION RESISTANCE

None of the finishes caused an undue loss in abrasion resistance when tested by the Taber Abraser<sup>2</sup> with the modifications in the test method as cited above.

## MILDEW AND ROT RESISTANCE

In the results of resistance to mildew and rot, aside from copper and mercury compounds, already known for this property, the performance of cobaltous chromate, cobaltous metaborate, and cobaltous hydroxide merit attention.

## RESISTANCE TO OUTDOOR WEATHERING AND ACTINIC DEGRADATION

Many compounds were found to offer resistance to outdoor weathering. This was the property wherein these experiments were most successful. Very good results were obtained with beryllium oxide, cobaltous hydroxide and lead chromate. Good results were obtained with cobaltous metaborate, cobaltous sulfide, **magnesium oxide**, manganous oxide, nickelous phosphate, and aluminum hydroxide.

It is interesting to note that some treatments were found to provide both mildew and rot resistance as well as resistance to outdoor weathering. These were compounds of cobalt, copper, and nickel. The resistance to actinic degradation using the Fade-Ometer was determined. The results, together with the results obtained from the weathering experiments are plotted in Figure 1. Poor correlation was ob-



tained between the results from the Fade-Ometer and from weathering. This lack of

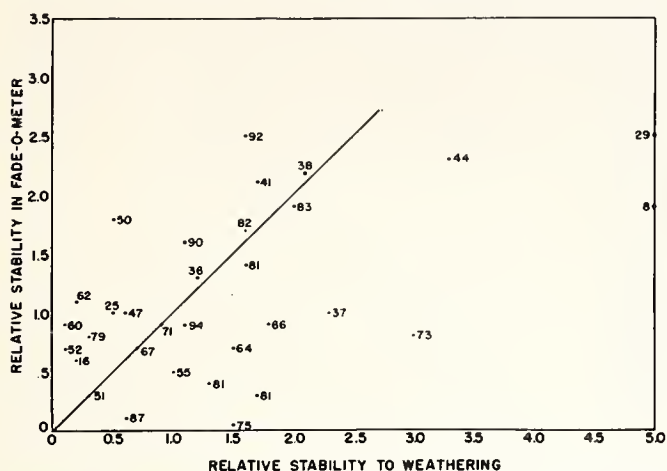


Figure 1.—Relation between the relative results obtained for outdoor weathering and the Fade-Ometer for treated cotton fabrics.

correlation had been determined previously by Fynn, Daly, Fleming, and Dean (88). Tests in the Fade-Ometer involve the exposure of the fabric to the carbon arc spectrum filtered by Corex glass, which is not identical to the spectrum of sunlight. In addition, the effects of atmospheric contaminants and water are included in weathering.

An attempt has been made to obtain a better laboratory method for predicting the outdoor weathering performance of the treated fabrics. Atmospheric contaminants have been neglected since the location of these experiments, Denton, Tex., is relatively free from industrial and other fumes.

Based on a general theory, weathering can be explained as a process of autooxidation of the cellulose. This process of autooxidation is catalyzed both by light and by heat, one or the other of which is necessary to excite the molecules. This is common to all autooxidation of organic compounds.

Microscopic investigations of the treated fabrics indicate that these inorganic finishes are deposited primarily on the surface of the fibers. In other words the finish acts as a filter which shields the cellulose from part of the light spectrum.

The effect of the finish, in a simplified explanation is twofold:

- 1) To shield the cotton from a portion of the light spectrum.

- 2) To inhibit oxidation by acting as a radical chain breaker. Some compounds, on the other hand, may catalyze the oxidation.

To give a quantitative value to the protection afforded by light screening by the different finishes, the relative damage caused by the different wavelengths of the spectrum had to be determined. In Figure 2, the data obtained

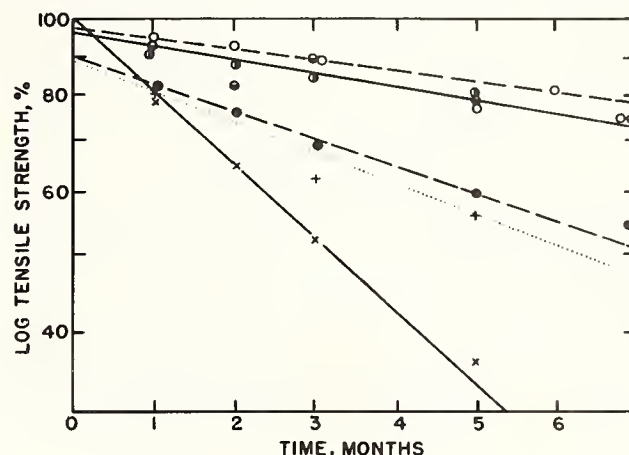


Figure 2.—Plot of the data of Fynn, Sands and Campbell (89) on the degradation of cotton fabric by filtered sunlight.

by Fynn, Sands, and Campbell (89) for degradation of a fabric exposed to sunlight under different filters was plotted on semilogarithm paper, with the logarithm of the tensile strength plotted against time. The different filters shielded the fabric from different regions of the light spectrum. The relative rates of degradation were calculated for the different filters. The degradation was found to follow first order kinetics. From the relative rates, the contribution of the different parts of the spectrum to weathering was calculated. The approximate values obtained were:

290-330 m $\mu$	35%
330-400	40
400-600	5
600-1000	5
Infrared	15

#### Key for Legend

Types of glass filters:	Wavelength transmitted: m $\mu$
X Corex	290 on
+ Blue	300-350,700 on
● Ultraviolet	300-400
O Infrared	1000 on
⦿ Yellow	550 on
● Red	600 on

From the relative damage caused by different wavelengths, and the relative absorptions in those wavelengths, the Index of Light Screening was determined. A sample plot is given in Figure 3. To determine the regions

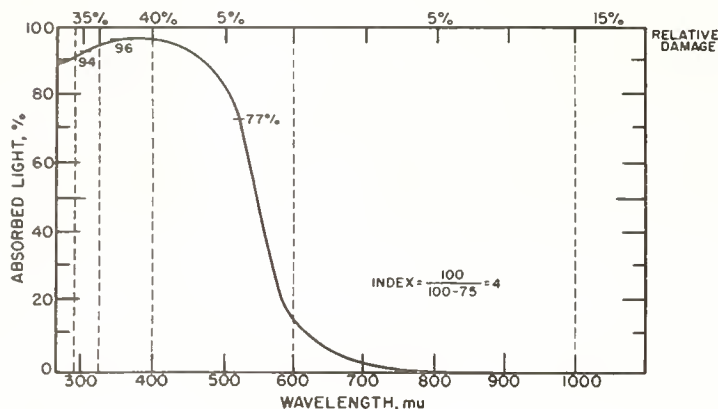


Figure 3.—Ultraviolet absorption of cotton fabric treated with 85 percent lead chromate.

of the spectrum screening by the compounds investigated, the absorption spectrum of certain compounds was obtained with the spectrophotometer, by making a mull on the side of the cell. Therefore, the light which would be screened away from the cellulose would not be available for catalyzing the degradation. The same results were obtained more readily by using the reflectance attachment and obtaining the reflectance of the treated fabric against the control. The light which was not reflected was considered to have been absorbed by the finish.

The second assumption made, which in the opinion of the authors is not so strong as the first, is that if the compound acts as an inhibitor to oxidation, or as a chain breaker, it would be effective both for autooxidation catalyzed by light and by thermal excitation. For this reason, the rates of degradation of the treated fabrics in the oven were determined.

Putting the two factors together, a theoretical relationship was advanced between weathering and the two factors of light screening and inhibition or catalysis of oxidation by the compound in the oven at 140° C. Table 7 gives the Light Screening Index (I.), the stability to aging in the oven (C.), the product of these two factors, and the stability to weathering given to cotton by different compounds.

There is good agreement in many cases between the product of the two factors of Light Screening Index and stability to aging in the oven and the weathering stability. The results are plotted in Figure 4, in which it is seen that

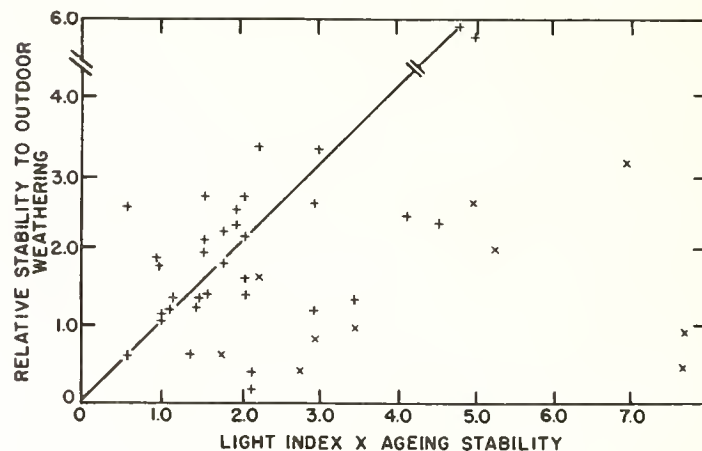


Figure 4.—Relation between the Light Screening Index and stability to oxidation in the oven at 140° C. with the stability of the treated fabric to weathering. Finishes that fail to follow the postulated relationship, whose deviation can be accounted for by instability when stored or by the pH of the fabric, X.

numerous compounds fall close to the theoretical line. Some of those where this relation fails can be explained by the fact that the fabric tenders on storage, or can be explained as a result of the acidity or basicity of the treated fabrics. For others, no explanation is given by this oversimplified picture for the reason that they fail to agree with the theoretical prediction. For one thing, oxidation at 140° C. in the oven is not the same as oxidation during outdoor weathering. In short, this postulated relationship holds good for a majority but not for all cases and gives much better correlation than the results obtained with the Fade-Ometer.

## RESISTANCE TO HEAT EXPOSURE

Only 5 treatments of exposure to heat in a 140° C. oven for 120 hours improved the resistance of cotton to degradation sufficiently to be considered. These were: Cerous hydroxide, cobaltous sulfide, manganous phosphate, lead chromate, and zinc phosphate.

Failure to improve this property in comparison with untreated cotton would not impair the suitability of a fabric for most end uses.

## FLAMMABILITY

Only 14 of the compounds improved cotton sufficiently at the add-on concentrations used to permit the treated fabric to pass the 45° angle flammability test. These were ceric oxide; chromic oxide; ferric hydroxide at both add-ons used; ferrous sulfide; lead chromate; lead hydroxide at the three add-ons studied, lead metaborate, and lead silicate; magnesium oxide at two levels of add-ons; manganous oxide; molybdenum oxide at both add-ons studied; tin (stannous) hydroxide, tin (stannic) oxide; and titanium dioxide.

Seven additional compounds rendered the cotton slower burning, although the treated fabric failed the 45° angle flammability test. All of the fabrics which passed the 45° angle test were subjected to the vertical flame test. The two which passed this test were: Ferric hydroxide (10.4 percent add-on), which was

included in the study for comparative purposes, and lead hydroxide (15.3 percent add-on).

## THERMAL STABILITY

Only 11 compounds at the add-on levels studied improved cotton fabric with respect to thermal stability. These were: Barium chromate; cupric oxide; magnesium oxide; manganous oxide and manganous silicate; and six lead compounds—the chromate, the hydroxide, the metaborate, the oxalate, the silicate, and the sulfide.

## WATER REPELLENCY

The water repellency was found to be improved for a number of compounds. The compounds scoring a spray rating above 50 and up to 70-80 were cobaltous metaborate, bismuth hydroxide, lead hydroxide, cobaltous sulfite, and cobaltous chromate.

## CONCLUSIONS

The results of this study show no distinct relationship which can be attributed to the position of the elements in the Periodic Table. The nine compounds (to be compared with lead chromate) which were chosen for further consideration, because of the favorable results they exhibited in certain of the screening tests, also showed a broad distribution in the Periodic Table.

The 10 compounds selected as a result of this study for further investigation to determine their suitability as recommended treatments for cotton were: Lead chromate (for comparative purposes), aluminum hydroxide, beryllium oxide, cobaltous hydroxide, cobaltous metaborate, lead hydroxide, manganous oxide, manganous phosphate, manganous silicate, nickelous phosphate.



# PART III—FURTHER EVALUATION OF NINE PROMISING INORGANIC COMPOUNDS

by

**Ramon M. Esteve, Jr., Georgia Ellen Wright, and Pauline Beery Mack**

## INTRODUCTION

## MATERIALS AND EXPERIMENTAL METHODS

Application of Finishes

Evaluation of Finishes

## RESULTS

Color of Treated Fabrics

Retention of Finish During Leaching and Laundering

Strength Losses Through Application of Treatment and During  
Darkroom Storage

Resistance to Mildew and Rot

Retention of Finish During Outdoor Weathering

Resistance to Weathering

Resistance to Actinic Degradation

Retention of Strength During Exposure to Atmospheric  
Contaminants

Resistance to Heat Degradation

Flammability

Thermal Stability

Water Repellency

## CONCLUSIONS



# INTRODUCTION

As a result of the exploratory study of the treatment of cotton fabrics with 74 inorganic oxides and salts (Part II of this report), nine compounds were selected as worthy of further study, because of one or more functional properties in which they improved the cotton fabric and because none of these compounds has been reported previously as finishes for cotton fabrics.

Six additional levels of add-on were used, involving 4 of the 9 compounds; laboratory tests were repeated on all of the treatments formerly reported (Part II); an integrated leaching-laundering test was used; and a test for resistance to atmospheric contaminants was introduced into the series.

Specifically, the evaluation techniques included the following: Retention of add-on during a combined leaching-laundering test; relative stability to mildew and rot according to the soil burial test; relative stability to outdoor weathering for 1, 2, and 3 months, respectively; retention of add-on after 3 months of outdoor weathering; relative stability to actinic degradation, (Fade-Ometer); retention of breaking strength after exposure to atmos-

pheric contaminants; relative stability to heat degradation; flammability test; relative thermal stability; and water repellency.

One of the principal properties that has been found to be improved in cotton by certain selected treatments is resistance to weathering. For this reason, the results of the tests applied in this study were evaluated in comparison with a compound known for its excellent results in improving weathering stability in cotton, lead chromate.

Studied by the Southern Utilization Research and Development Division of the United States Department of Agriculture, lead chromate has lengthened the life of cotton fabric greatly, by increasing the fabric's stability to weathering (54, 251), as shown by its application to tobacco shade cloth. The term weathering refers to the complex deterioration of a fabric as a result of outdoor exposure. More precisely, the various deteriorating factors include such agents as actinic degradation (more accurately described as the autooxidation of the cellulose catalyzed by solar radiation), the effect of atmospheric contaminants, and microbiologic degradation (292).

## MATERIALS AND EXPERIMENTAL METHODS

### APPLICATION OF FINISHES

The finishes were applied as described in Part II. Additional levels of add-on, used for the first time in this unit of the series, were applied as follows:

For aluminum hydroxide the level of add-on employed in Part II was employed first, and two additional levels were employed also. Likewise, two additional levels of cobaltous hydroxide and one each of cobaltous metaborate and lead hydroxide were employed.

The methods of application of these additional treatments (with the colors resulting) are listed below:

**Aluminum hydroxide** (add-on 3.6 percent).

First bath—10 percent aluminum acetate in water with 2.5 percent acetic acid. Second bath—15 percent sodium carbonate at 70° C., cured at 120° for 20 minutes. Color: white.

**Aluminum hydroxide** (add-on 5.2 percent).

First bath—12 percent aluminum acetate in water with 2.5 percent acetic acid. Second bath—15 percent sodium carbonate at 70° C. Cured at 120° for 20 minutes. Color: white.

**Cobaltous hydroxide** (add-on 5.6 percent).

First bath—8 percent cobaltous acetate. Second bath—5 percent sodium hydroxide at 70° C. Rinsed in hot water. Color: tan.

**Cobaltous hydroxide** (add-on 8.2 percent).

First bath—10 percent cobaltous acetate. Second bath—6 percent sodium hydroxide at 70° C. Color: tan.

**Cobaltous metaborate** (add-on 8.9 percent).

First bath—6 percent cobaltous acetate. Second bath—10 percent sodium metaborate. Fabric kept in second bath 35 minutes at 70° C. Color: gray.

**Lead (plumbous) hydroxide** (add-on 6.5 percent). First bath—10 percent lead acetate. Second bath—10 percent ammonium hydroxide at 70° C. Cured at 120° for 20 minutes. Color: white.

### EVALUATION OF FINISHES

The tests used to evaluate the selected nine finishes were described in Part II of this series: Breaking, bursting, and tearing strengths; abrasion resistance; mildew and rot resistance by the soil burial method; resistance to outdoor

weathering; resistance to actinic (Fade-Ometer) degradation; flammability; thermal stability; and water repellency. The leaching and laundering techniques were administered in this study according to the same procedures as those described in the screening report (Part II), although a composite test was used in which two leaching procedures were followed by one laundering. The test for resistance to atmospheric contaminants was that developed by Ray (223) following an evaluation by Ray, Mack, and Wachter (224).

## RESULTS

**Table VIII embodies the major findings of this study.**

### COLOR OF TREATED FABRICS

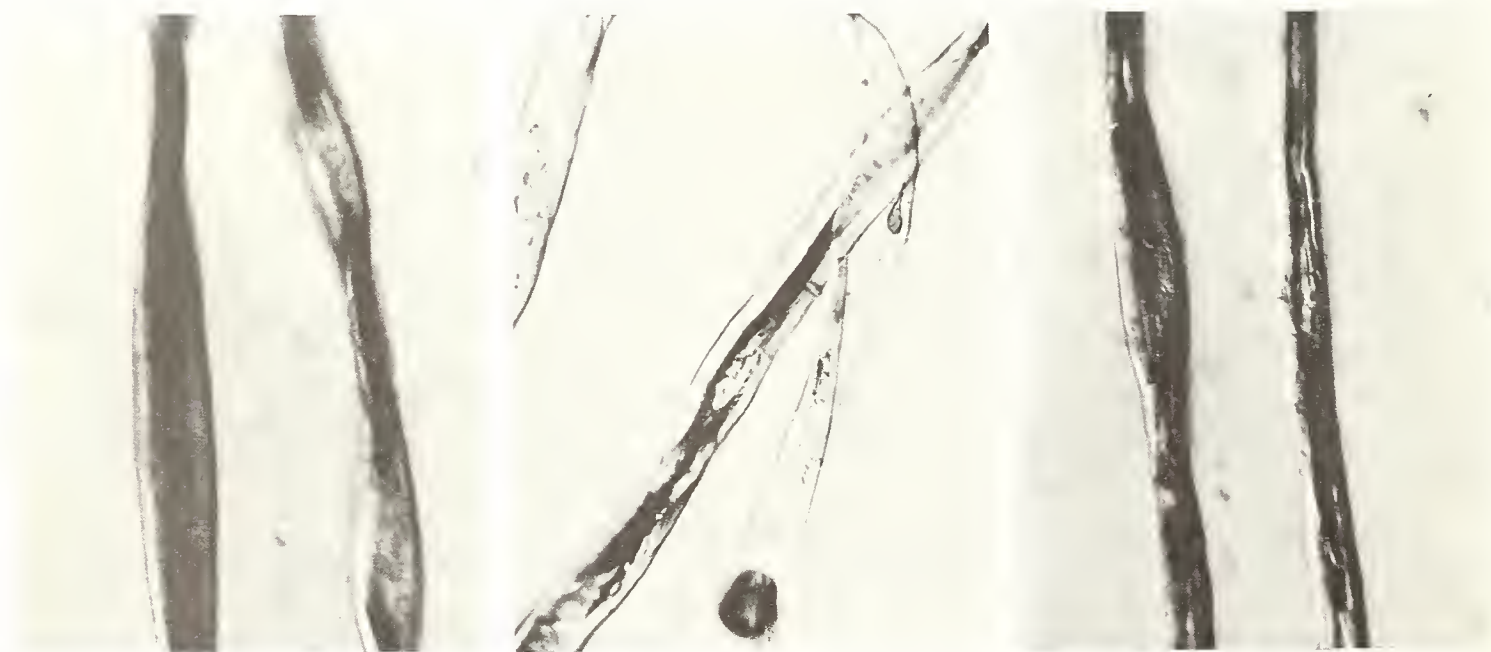
Cotton swatches treated with the following compounds were found to be white: Beryllium oxide, aluminum hydroxide, lead hydroxide, and manganous phosphate. Nickelous phosphate treated cotton is cream; manganous silicate cotton is tan; cobaltous metaborate cotton is gray; cobaltous hydroxide cotton is light tan changing to light brown upon partial oxidation; and manganous cotton is oxide brown.

Change of the tan color of cotton treated with cobaltous hydroxide can be brought about by oxidizing with cold 0.5 percent sodium hypochlorite. The partially oxidized cobaltous hydroxide has the additional advantage of

being more resistant to laundering and to dilute acids.

### RETENTION OF FINISH DURING LEACHING AND LAUNDERING

The retention of the finish, determined after 2 leachings followed by 1 laundering, ranged from 50 to 97 percent. The greater proportion of the loss of the finish occurred during the first leaching. This brought up the question of penetration. Cotton fibers treated with lead chromate and with cobaltous hydroxide were therefore examined microscopically. There appeared to be some penetration into the fiber, although the bulk of the inorganic compound was deposited on the surface of the fiber, as shown in Figure 5.



**Figure 5.**—Photomicrographs of cotton treated with lead chromate (left), and cobaltous hydroxide (right) X 900. The similarity of the two samples and the contrast with the control (center) is apparent.

Table VIII.—Responses of cotton fabrics treated with 9 selected inorganic compounds

Fabric Treatment	Relative Stability to Outdoor Weathering					Percent add-on	Color of treated cotton	Percent add-on retention after 2 leachings and 1 laundering	Relative stability to mildew and rot (soil burial method) in days	Relative Stability to Outdoor Weathering				Percent retention of wet breaking strength after exposure to atmospheric contaminants	Relative stability to heat degradation (oven at 140° C. for 5 days)	Response to flammability test—45° angle flame test; vertical flame test for fabrics passing the 45° angle test	Relative thermal stability	Water repellency (spray rating)
	After 1 month	After 2 months	After 3 months	Percent retention of add-on after 3 months of outdoor weathering	Relative stability to actinic degradation (Fade-O-meter at 53° C.)													
Untreated cotton	0.0	white	1+	1.0	1.0	1.0	1.0	1.0	1.0	29	1.0	1.0	Burned with afterglow 13 seconds	1.0	0			
Lead (plumbous) chromate (for comparative purposes)	8.5	orange	72.0%	1+	7.0	8.7	4.6	86%	1.9	1.5	85	1.5	Passed 45° angle test. Failed vertical flame test	2.0	0			
Aluminum hydroxide	3.6	50	5	2.0	1.6	1.8	1.8	81	1.6	1.0	81	1.0	Passed 45° angle test Failed vertical flame test	0.3	0			
	5.2	white	69	4	2.0	2.0	2.0	100		1.1	90	1.1	Failed—burned with afterglow 48 seconds		0-50			
	8.7	57	5	2.0	1.6	2.4	2.4	94			90		Failed—burned with afterglow 47 seconds		0-50			
Beryllium oxide	1.9	white	80	5	3.0	3.3	4.2	100	0.9	0.6	100	0.6	Failed—burned with afterglow 38 seconds	0.2	0			
Cobaltous hydroxide	4.5	75	14	3.8	5.4	4.1	4.1	80	2.5	1.1	100	1.1	Failed—burned with afterglow 13 seconds	0.7	0			
	5.6	tan (light brown when oxidized)	70	16	10.0	4.7	6.0	90	3.4	1.2	100	1.2	Failed—burned with afterglow 13 seconds	0.7	0			
	8.2	95	6	10.0	7.1	7.1	7.1	100	3.4	1.2	100	1.2	Failed—burned with afterglow 13 seconds	0.7	0			
	16.2	97	8	5.4	5.3	5.3	5.3	95			95				0			
Cobaltous metaborate	6.0	gray	74	16	2.0	2.7	3.3	100	3.3	0.9	90	0.9	Failed—burned with afterglow 15 seconds	0.9	50			
	8.9	85	18	8.3	4.7	4.5	4.5	100		0.5	96	0.5	Failed—burned with afterglow 15 seconds	0.9	70-80			
Lead (plumbous) hydroxide	4.4	50	2	0.9	0.8	1.0	1.0	97	0.6	0.6	97	0.6	Passed 45° angle test	1.5	70			
	6.5	white	80	1+	1.0	1.0	1.0	98		1.2	98	1.2	Passed 45° angle test		50			
	8.4	80	1+	1.0	1.0	1.0	1.0	90		1.2		1.2	Passed 45° angle test		50			
	15.3	75	1+	1.1	1.1	1.0	1.0	88	1.0		100		Passed 45° angle test. Passed vertical flame test	1.1	70			
Manganous oxide	14.5	red-brown	86	2	2.2	2.3	2.0	96	2.2	0.6	92	0.6	Passed 45° angle test	1.2	0-50			
Manganous phosphate	4.0	white	92	1+	2.2	1.6	1.8	88	1.9	1.3	58	1.3	Failed—burned with afterglow 50 seconds	0.8	0			
Manganous silicate	7.0	pale tan	90	1+	2.2	1.4	1.4	70	1.7	1.0	70	1.0	Failed—burned with afterglow 50 seconds	1.3	0			
Nickelous phosphate	5.1	cream	60	4	1.2	2.0	3.2	56	1.4	0.6	65	0.6	Failed—burned with afterglow 50 seconds	0.8	50			



The fact that most of the finish is on the surface of the fiber accounts for most of the loss of add-on during the leaching-laundering treatment. Since the loosely-held portion of the deposit could be washed away easily, the greater portion of the loss occurred during the first leaching.

### STRENGTH LOSSES THROUGH APPLICATION OF TREATMENT AND DURING DARKROOM STORAGE

None of the cotton swatches treated with the compounds of this study lost notable percentages of breaking, bursting, or tearing strengths, or of abrasion resistance, either as a result of the finish or during subsequent darkroom storage.

### RESISTANCE TO MILDEW AND ROT

The resistance to mildew and rot is reported as the time in days required for the fabric to lose one-half of its wet strength in the soil burial test. Lead chromate, lead hydroxide, manganous oxide, manganous silicate, and manganous phosphate performed approximately the same as untreated cotton specimens in this respect. Beryllium oxide, aluminum hydroxide, and nickelous phosphate exhibited slight improvement in stability to deterioration by this means, while cobaltous metaborate gave good, and cobaltous hydroxide moderate to good resistance to this deteriorating agency. The lower percentages of add-on of the latter treated fabric surpassed the higher add-ons.

### RETENTION OF FINISH DURING OUTDOOR WEATHERING

The retention of add-on during 3 months of outdoor weathering ranged from 56 to 100 percent. Nickelous phosphate treated cotton retained only 56 percent of the finish during the 3 months of outdoor weathering; manganous silicate retained 70 percent; all other of the experimental fabrics retained more than 80 percent. Many of these cottons retained during 3 months of outdoor exposure more than 90 percent of the add-on.

Retention of finish during 3 months of outdoor weathering surpassed that during leaching and laundering in all but two cases, manganous phosphate and nickelous phosphate. In both cases the difference between the two treatments was slight.

### RESISTANCE TO WEATHERING

Cobaltous hydroxide (add-ons 5.6 and 8.2 percent) and cobaltous metaborate (add-on 8.9 percent) afforded protection from weathering to cotton fabrics which was similar to that imparted by lead chromate. Beryllium oxide in the respect of weathering approached the performance of lead chromate. Aluminum hydroxide, manganous oxide, manganous silicate, manganous phosphate, and nickelous phosphate also offered some resistance to weathering, although they were less effective than either lead chromate or the two cobaltous compounds. Lead hydroxide did not enhance the weathering stability of cotton.

The deterioration of cotton fabrics from weathering was found to behave kinetically as a first order process. Figure 6 gives the

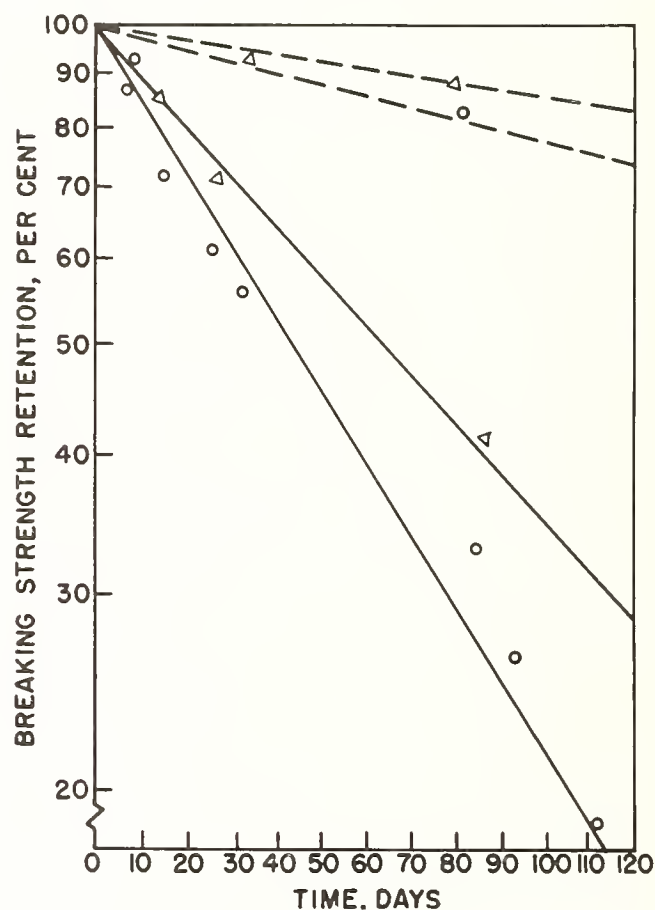


Figure 6.—Retention of wet (o) and dry ( $\Delta$ ) breaking strength after exposure to outdoor weathering.

Untreated Cotton —————  
Treated Cotton - - - - -

strength retention (dry and wet) of a representative trial. The results are reported as the stability to weathering obtained from the reciprocal of the relative rate of breaking



strength loss. Both wet and dry breaking strength gave comparable results. Wet strength measurements give the desired information in less time than dry strength measurements, because the rate of wet strength loss is more rapid than that of dry strength loss with cellulose fibers or fabrics.

## **RESISTANCE TO ACTINIC DEGRADATION**

Relative stability to actinic degradation as determined by exposure in the FDAR Fade-Ometer for a series of trials up to 300 hours at 53° C, was 1.9 for cotton treated with lead chromate. This approximate relative stability was shown by the aluminum hydroxide, the manganous phosphate, and the manganous silicate treated cotton fabrics. A slightly greater relative rate of degradation was obtained for manganous oxide treated fabrics; a lesser value was obtained for cotton fabric finished with nickelous phosphate. Beryllium oxide and lead hydroxide treated cottons gave slightly poorer results than were obtained with the unfinished cotton.

The highest relative stability to actinic exposure based on breaking strength was obtained with the cotton treated with cobaltous metaborate and cobaltous hydroxide.

## **RETENTION OF STRENGTH DURING EXPOSURE TO ATMOSPHERIC CONTAMINANTS**

All of the treated fabrics considered in this study retained their wet breaking strength when exposed to atmospheric contaminants to a greater degree than did the untreated cotton, which had a retention only of 29 percent. The treated fabrics ranged from 58 percent strength retention (manganous phosphate) to 100 percent for 3 of the treatments, including cobaltous hydroxide at the 3 lower levels of add-on. The three highest strength losses were with manganous phosphate, already cited, nickelous phosphate (65 percent retention), and manganous silicate (70 percent retention).

## **RESISTANCE TO HEAT DEGRADATION**

Relative heat stability of the untreated and treated cotton fabrics was calculated from

changes in breaking strength. Lead chromate, cobaltous hydroxide (5.6 and 8.2 percent add-ons), lead hydroxide (6.5 and 8.4 percent add-ons), manganous phosphate treated cottons slightly exceeded untreated cotton in this test, with the remainder little or no better than, or even slightly below the untreated fabric. There probably would be few end uses for which resistance to heat degradation would be a factor.

## **FLAMMABILITY**

In the test for the flammability of the treated fabrics, lead chromate, aluminum hydroxide (add-on 3.6 percent), lead hydroxide (at 4 levels of add-on), and manganous oxide passed the 45° angle test. Of these, only the cotton treated with lead hydroxide with an add-on of 15.3 percent passed the vertical flame test.

All other treated fabrics in this study burned in the 45° angle flame test, but several burned more slowly than did the untreated cotton, as shown in Table VIII.

## **THERMAL STABILITY**

The thermal stability of cotton treated with lead chromate was twice that of untreated cotton. Lead hydroxide, manganous oxide, and manganous silicate increased the thermal stability of cotton, but to a somewhat less degree. Aluminum hydroxide and beryllium oxide decreased the thermal stability of cotton notably, while cobaltous hydroxide, cobaltous metaborate, manganous phosphate, and nickelous phosphate decreased this factor slightly. Cobaltous metaborate treated cotton had practically the same thermal stability as untreated cotton.

## **WATER REPELLENCY**

Cottons treated with cobaltous metaborate and lead hydroxide displayed good water repellency, while some water repellency was imparted by aluminum hydroxide (8.7 percent add-on), manganous oxide, and nickelous phosphate. Cotton fabric treated with lead hydroxide was not water repellent unless the fabric was cured at 120° C. for 20 minutes after the application.

## CONCLUSIONS

The improvement in certain properties of cotton by the use of 9 inorganic compounds, selected from a screening study involving 74 compounds and subjected to more intensive study, seems sufficient to warrant investigation of the 9 compounds under practical conditions. The finishes producing white or cream textiles might prove suitable as household fabrics because some of them demonstrated improved resistance to weathering and to atmospheric contaminants. Some of them, in addition, were found to promote resistance to atmospheric contaminants, some degree of flame resistance, and some water repellency. Lead hydroxide finished cotton was a white fabric which differed from the other whites in having no resistance to weathering, although it was found to have very good resistance to atmospheric contaminants. This fabric had the added virtue of possessing the best resistance to burning of any of the nine experimental compounds, to-

gether with good to very good water repellency.

The tan, gray, and brown treated cotton could find application in various agricultural end uses. Among these experimental fabrics, cobaltous metaborate treated fabric should warrant especial attention in further practical trials because of its excellent performance in the tests for mildew and rot, weathering, resistance to actinic degradation, stability with respect to atmospheric contaminants, and water repellency. Cobaltous hydroxide was similar in performance except for negative results in the water repellency test. The manganese and nickel compounds used, which gave colored treated cottons, were far from as outstanding as cobaltous hydroxide or metasilicate, although they made minor contributions in some respects.

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